

LABORATORY MANUAL

W. LITTLER

QD

33

L77

1934

LAB•MAN•

C. 2

CURR

CONTENTS

	Page		Page
"Safety First" Rules ..	1	21. Sodium Hydroxide	37
1. Manipulation of Glass Tubing	2	22. Other Compounds of Sodium	39
2. Other Simple Opera- tions	4	23.* Atomic Weight of Sodium	41
3. The Balance and Weighing	7	24.* Formula of Glauber's Salt	43
4. Methods of Measuring Volumes of Liquids	9	25. Properties of Sodium Thiosulphate	44
5.* Law of Multiple Pro- portions	11	26.* Preparation of Potas- sium Chlorate	45
6. Reversible Reactions ..	12	27. Density of Oxygen and Percentage of Oxy- gen in Potassium Chlorate	46
7.* Raoult's Law	14	28. Potassium Nitrate	48
8. Ionization	15	29. Preparation of Potas- sium Bromide and Iodide	50
9. Hydrolysis	16	30.* Potassium Carbonate from Wood-ash	52
10. Neutralization-(I)	17	31.* Hardness of Water	54
11.* Neutralization-(II)	22	32. Some Compounds of Magnesium	56
12.* Neutralization-(III)	23	33. Valency of Magnesium	57
13. Electrochemical Series	25	34. Zinc and some of its Compounds	60
14.* Hydrogen Fluoride	26	35. Aluminium and some of its Compounds	62
15. Hydrogen Bromide and Bromine	27	36. Iron and some of its Compounds	64
16. Hydrogen Iodide and Iodine	29		
17. Chlorides, Bromides and Iodides	31		
18.* Calcium Phosphate from Bones	33		
19. Arsenic and some of its Compounds	34		
20. Antimony and some of its Compounds	36		

(Contents continued on inside back cover)

LABORATORY MANUAL

to accompany "Elementary Chemistry — II"

BY

W. LITTLER, B.A., B.Sc.

Senior Science Master at Hele's School, Exeter

Author of "Elementary Chemistry"

T O R O N T O

CLARKE, IRWIN & COMPANY LIMITED

1943

COPYRIGHT, CANADA, 1934
by CLARKE, IRWIN & COMPANY LIMITED

First Published, November, 1934
Reprinted, August, 1936
Reprinted, October, 1938
Reprinted, June, 1940
Reprinted, August, 1941
Reprinted, July, 1943

MIMEOGRAPHING OR REPRODUCING MECHANICALLY IN ANY OTHER WAY PASSAGES FROM THIS BOOK WITHOUT THE WRITTEN PERMISSION OF THE PUBLISHERS IS AN INFRINGEMENT OF THE COPYRIGHT LAW

LIBRARY
UNIVERSITY OF ALBERTA

Printed in Canada

INTRODUCTION

THIS little book has been written primarily as a companion volume to the author's "Elementary Chemistry, Book II", but it will probably be found suitable for students taking the more advanced part of their Chemistry course even if they are using some other book for their theoretical work.

In the selection of material, the writer has kept in mind the fact that, for various reasons, some schools can attempt more than others in the way of practical work. Further, it often happens that there are two or three enthusiastic students in a class who, with a little help from their teacher, are willing and anxious to do practical work which is considerably beyond the bare requirements of the curriculum. A number of exercises have, therefore, been included (Ex. 7, for example) which can be omitted by schools that are unable to attempt much more than the necessary minimum. Such exercises are marked with an *asterisk*. Some of the remainder, though presenting no special difficulty, may be found to contain more material than can be worked through in the time available. Ex. 35 on p. 62 is an example. In such cases, one or more of the numbered paragraphs (which are usually independent of one another) may be omitted by everybody. Alternatively — and this is the writer's own practice — different paragraphs may be worked through by different pupils, so that the whole is covered. It is not hard to arrange that each student shall see each experiment, at least in its culminating phase, and all the varied material can be linked together in the "discussion" which will follow the practical work — if not in the laboratory, then in the next "theory" lesson.

It is assumed that a close relation between practical and theoretical work will be maintained throughout. This places on the teacher the considerable burden of making sure that for each lesson the necessary apparatus and materials are ready. To lighten this task as far as possible, a list is given (Appendix II, p. 100) of the apparatus

which each student should have in his cupboard and of the materials he should have on his bench. This regular stock is referred to in the various exercises as "*Apparatus and Materials.—(a)*". In arranging for the lesson, the teacher may ignore these and confine his attention to "*Apparatus and Materials.—(b)*". A list of most of the solutions mentioned here, with directions for making them up, will be found in Appendix IV. A few solids (manganese dioxide, ferrous sulphate, etc.) are also included in that list.

Obviously it is important to make sure, before the beginning of the school year, that all necessary apparatus and materials are available, and Appendix VI (p. 104) gives a list of what would be required for 10 students.

The work of preparing apparatus is, of course, much reduced if the students work in pairs, and indeed there are sound arguments for such an arrangement, quite apart from that of convenience. In cases, however, where the practical operations are of a very simple character (*e.g.* involving only the use of test-tubes), the students should work singly.

Most teachers require each student to write up his notes; but time is short, and one must not expect too much. In the "*Directions, etc.*" for each exercise will be found letters "*Q.1.*", "*Q.2.*", etc. indicating (generally) a question to be answered, or in any case some note to be made. If the "answers" are numbered it will greatly reduce the work of marking.

In most of the Exercises it will be noticed that *quantities* are mentioned. Thus, in Ex. 21 (preparation of sodium hydroxide) the weights of washing soda and slaked lime, and the volume of water are given, as well as the time for which the mixture should be boiled. It will be understood, of course, that in such cases the quantities mentioned are only intended to be approximate. It is the writer's experience, however, that many experiments "go wrong" simply because the quantities have been badly chosen, and in an ordinary school course there is rarely

time to repeat an exercise. He has, therefore, tried out the various experiments under different conditions, noting the quantities which gave the best results. It takes very little longer to make a rough weighing (say within 10 per cent.) of the substances used, and there is full compensation in the fact that one can depend on successful results if the directions are followed.


Alternatively (still quoting Ex. 21 for illustration), the teacher could arrange for a specimen 20 gm. of washing soda to be weighed out and put on a piece of paper on the demonstration bench; similarly 10 gm. of slaked lime, and 150 c.c. of water in a beaker. The students can then see at a glance just what quantities to take.

Some of the exercises, however, are definitely quantitative and, needless to say, in such cases weighing and measuring must be carried out with as much accuracy as possible.

A word must be said on the subject of accidents. Here prevention is much better than cure, and the writer has found it helpful to direct the attention of his own classes to the "Safety First" Rules which appear immediately after this Introduction. Appendix I (p. 98) contains a few "First Aid" directions, and the questions which follow them bear partly on "First Aid", but chiefly on the "Safety First" rules. It is hoped that these questions may serve as the basis for an occasional class discussion, and may help to make sure that the rules are attentively read.

W. LITTLER

HELE'S SCHOOL,
EXETER, ENGLAND,
July, 1934.



Digitized by the Internet Archive
in 2017 with funding from
University of Alberta Libraries

"SAFETY FIRST" RULES

1. Do not go into the laboratory without the permission of a teacher.
2. Do not perform any experiment, or mix or handle any materials, without the permission of the teacher in charge.
3. When heating a test-tube, see that it is not pointed towards yourself or anybody else.
4. If you get any acid on your skin, wash it under the tap at once.
5. When rinsing out a flask, retort, etc., which has contained strong acid, hold it at arm's length.
6. When pushing glass tubing through a rubber stopper, take a short hold. If the tubing is tightly stuck, ask the teacher to help you.
7. If the stopper of a bottle sticks, it may finally come out with a jerk, causing the liquid to splash into your face; so turn the bottle slightly away from you.
8. Except when there are instructions to the contrary, use *small* quantities of material. "As much as would go on a ten-cent piece" is a good general rule.
9. If when you are preparing a gas, acid begins rising rapidly towards the top of the thistle funnel, it usually means that the rubber connection is pinched. In releasing it, take care to stand as far away as possible.
10. Always attend very carefully to any special instructions given, either in the book or by your teacher. Failure to do this may result in an accident.

EXERCISE 1

MANIPULATION OF GLASS TUBING

Object.—To learn how glass tubing should be cut, bent, etc., and how a cork should be bored.

Apparatus and Material.—(a) Bunsen burner. (b) 18 ins. of glass tubing of external diam. about 6 mm.; $1\frac{1}{2}$ in. rubber tubing; 500 c.c. flask preferably of conical shape; 2-holed rubber stopper, or bark cork, to fit flask; triangular file; flame spreader to fit bunsen; ruler; flask; set of cork borers.

Directions, etc.—**Cutting.** Cut a $4\frac{1}{2}$ -inch length from the glass tubing as follows. Make *one* good file-scratch (do not “saw”). Grip the tubing, the thumbs being on either side of the scratch, but the latter being turned away from you. Now snap the tubing across, the snap-



FIG. 1.

ping movement being accompanied by a pulling one. The tube should break “clean”. In the same way, cut the remaining $13\frac{1}{2}$ in. of your tubing into lengths of $9\frac{1}{2}$ in. and 4 in.

Bending. Fit the flame spreader over your bunsen, make the flame luminous, and proceed to bend the $4\frac{1}{2}$ -inch piece nearly in the middle ($2-2\frac{1}{2}$ in.) to an obtuse angle (as in fig. of wash bottle, p. 4). To do this, lay it so that the part to be bent is *along* (not across) the flame, twirling it round. This secures fairly even heating for a length of two inches or so. When the tube begins to bend, remove the left hand, take the tube out of the flame and



FIG. 2.—Bending.

let it bend to the correct angle by its own weight, checking it with the left hand if it is going too far. In the same way bend the $9\frac{1}{2}$ -inch piece to the acute angle indicated in Fig. 4 the bend being about $1\frac{1}{2}$ inches from one end.

Rounding Off. At present the ends of the glass are sharp and would cut into rubber tubing,—or your fingers. To “round off”, first move the end portion of the glass to and fro in the non-luminous flame (without the spreader) for 5 or 10 seconds. After this preliminary warming, push the end just into the edge of the flame, about 2 in. from the top of the burner, gently rotating the tube. The flame soon acquires a strong yellow tinge (due to sodium compounds in the glass). A few seconds later the tube may be withdrawn. Round off the ends of all the tubing in the same way.

Drawing Out. Take the 4-inch piece and holding it with one end in each hand, heat it in the ordinary non-luminous flame, with constant turning, round about a point $1\frac{1}{2}$ inches or so from one end.¹ When the heated portion is soft, remove the glass from the flame, and pull gently outwards. When the tube is cool, scratch gently at F with the file, and then break it across at that point. The shorter piece will serve for the jet of the wash-bottle.



FIG. 3.

Putting Tube Through Rubber Stopper. The various pieces may now be assembled to make the wash-bottle of Fig. 4. In passing glass tubing through the hole of a rubber stopper (i) first run water through the hole; (ii) use a screw-like motion (do not simply *push*); and (iii) take a short hold, *i.e.* do not have your hand far from the stopper. Neglect of these precautions has often resulted in nasty accidents.

Connecting. When connecting the acute-angled piece to the jet, remember that rubber tubing is always much easier to fit over glass if it is first wetted.

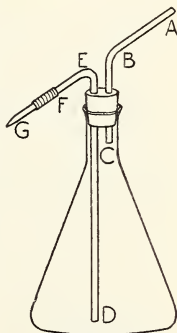


FIG. 4.—AB=2.5",
BC=2", ED=8",
EF=1.6", FG=1.8".

¹ It may be necessary to grip the short end with the crucible tongs.

Boring a cork. Rubber corks are far more satisfactory than bark ones, and their longer life soon makes up for the extra cost. However, if a bark cork is used in making the wash-bottle, it should be bored as follows.

First *soften* the cork by rolling it under the foot with steady (but not too heavy) pressure.

Choose a cork-borer of diameter slightly less than that of the glass tube to be inserted, and begin boring with a screw-like movement from the narrow end. Take great care to keep the borer parallel to the axis of the cork. When the borer is seen to be just about to break through the other end, take it out and finish the hole from the broad end.

If it should be necessary to make a hole through a rubber stopper, the borer should be first dipped in glycerine.

EXERCISE 2

OTHER SIMPLE OPERATIONS

Object.—To learn how to carry out a number of simple operations that often occur in practical chemistry.

Apparatus and Materials.—(a) *Two beakers; evaporating dish; glass rod; test-tube; retort stand with ring; funnel; wire gauze; tripod stand; bottle of lime-water.* (b) *Flask; filter paper; strip of paper about 4" \times $\frac{3}{4}$ "; sand; liquid consisting of soot ground up with water in a mortar (so as to wet it thoroughly) to which a quantity of brine has been added.*

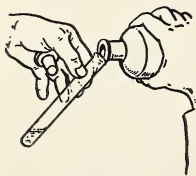


FIG. 5.

Directions, etc.—*Pouring from bottle to test-tube.* The point here is that the stopper of the bottle should not be laid down on the bench; otherwise stoppers soon become mixed.

Take the bottle (say the limewater bottle) in the right hand, holding the test-tube with the thumb and forefinger of the left. Grip the stopper

between the remaining fingers of the left hand and the palm, and when pouring let the mouth of the bottle press gently against the test-tube (to prevent drops of liquid running down the outside of the bottle).

The simple operation just described is such an extremely common one that it is worth while learning to carry it out properly.

Other cases of pouring. When pouring a liquid from one vessel to another a glass rod is generally used, being pressed against the first vessel to prevent the trickling of liquid down the outside (cf. Fig. 7). You will practise this presently.

Boiling a liquid in a test-tube. A common trouble is that owing to the production of vapour inside the liquid, some of the latter may be thrown violently out of the test-tube. This tendency is much reduced by (i) continually *shaking* the liquid with a rather rapid wrist movement; (ii) avoiding large quantities of liquid. A third of a test-tube is quite enough for most purposes.



FIG. 6.

In any case a good rule is:—*See that the test-tube is pointing neither towards yourself nor anybody else.*

Don't let the flame get to a part of the test-tube above the liquid. If you do, the test-tube becomes unequally heated and will very likely crack.

A very convenient holder for a test-tube is made by folding a piece of paper into a strip measuring about $4'' \times \frac{3}{4}''$.

Boiling a liquid in a beaker or flask. This is conveniently done by placing the beaker, etc. on a wire gauze supported by a tripod stand. The commonest causes of cracking are:— (i) letting the flame get above the level of the liquid, perhaps owing to the gauze being carelessly placed (ii) applying heat when there is a large amount

of solid material at the bottom (*e.g.* when preparing a solution of copper sulphate, or when heating hydrochloric acid with manganese dioxide). The explanation of the trouble belongs to the subject of convection, and is left for you to think out for yourself.

Decanting. This is a useful method of washing a heavy, insoluble substance, providing the latter settles readily. To illustrate the process, put a handful of sand in a flask and add water. Make the liquid swirl round a few times, then let it settle and pour off the water. After this has been done several times, any soluble impurities and any light insoluble ones are removed. The usefulness of decantation is rather limited, owing to slow settling. It is worth noting, however, that many powders, precipitates, etc. settle quite easily after the liquid in which they are suspended has been well boiled.

Filtering. Filtration, like decantation, is used to separate an insoluble solid from a liquid, but we do not wait for the solid to settle.



Place a funnel in the ring of your retort stand. Fold a filter paper exactly into halves, and then into quarters, pressing the creases, and open it into a cone shape, with three thicknesses on one side and one on the other. Now push it into the funnel, letting a little water fall on it to help it to grip. Press the paper gently against the glass. The more closely it fits, the more rapidly will it do its work.

Now stir up some of the brine and soot mixture and pour it down a glass rod as shown; the brine filters through and is collected in an evaporating dish placed below.

FIG. 7.—Filtering. N.B.—A hot (and especially a boiling) liquid usually filters faster than a cold one.

Evaporating. To illustrate this, place the dish containing the brine on a wire gauze supported by a tripod stand, and boil down to dryness.

When most of the water has been driven off, the wet salt will begin to "spit" a good deal. The trouble can be reduced by lowering the flame and taking it away from time to time, but the only real cure is to finish the evaporation by means of a water-bath, say by allowing the dish to rest on a beaker in which water is being boiled. This is a slow process, but in certain cases we have to resort to it, *e.g.* when the exact weight of dissolved salt has to be found.

EXERCISE 3

THE BALANCE AND WEIGHING

Object.—To examine a balance and a box of weights, and to learn how to weigh.

Apparatus and Materials.—(b) *Balance, weights, small objects to be weighed (pocket-knife, coins, etc.).*

Directions, etc. — Lift the side A from the hook B, and then lift off the hook itself. Put these to your left hand. Similarly, remove C and D, putting them to your right (so that later you can put them back to the side from which you took them). Now lift off the beam at E.

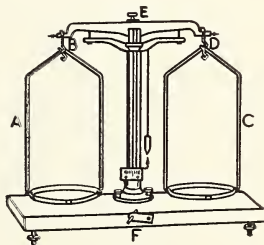


FIG. 8.

Notice the *knife-edges* on which the hooks rest. Notice also a third knife-edge at the middle of the beam. When a lever (sometimes a screw) at F is turned, a little platform is raised into contact with the knife edge at E, and the balance is then in action.

It is important to realize that *the sensitiveness of a balance depends on the knife edges remaining sharp*. The best knife edges are made of agate, but these are easily blunted by being chipped, if the balance is carelessly handled. If the following simple rules are observed, the knife edges will always remain sharp.

(1) Never put a weight on a balance pan, or take one off, except when the lever is down. The same rule applies, of course, with regard to the object to be weighed.

(2) Do not move a balance unless absolutely necessary. If you have to move it, make sure that the lever is down, and let your thumbs rest lightly on the pans to prevent them from swinging.

Now examine the box of weights, especially the fractions of a gram. These are usually 500 mgm., 2 — 200's, 100, 50, 2 — 20's, and 10 (smaller weights than 10 are often supplied, but may be ignored). Learn to recognize these weights by their *shape*. The 500 and 50 are hexagonal, the 200 and 20 are square, and the 100 and 10 are triangular.

Now weigh a small object, say a pocket-knife, proceeding as follows.

Put the object in the *left*-hand pan (unless you are left-handed) and by one or two trials find the two weights between which the true weight lies, *e.g.* suppose the object weighs more than 20 gm. but less than 50 gm. Leave the 20 gm. on the pan, and add the 10 gm. This (say) is too heavy. Take it off and put on the 5 gm. Suppose the weights are now too light. Leave the 5 gm. and add the 2 gm., and so on, trying all the weights in turn until you have worked down to the 10 mgm. weight.

To add up the weight correctly

- (i) make a list of the weights as they lie on the pan;
- (ii) check your list as you put them back into the box, always putting them into their right places. Then add up.

After a very little practice you will be able to

- (i) add them up mentally as they lie on the pan (writing down the total);

(ii) add them up as you put them back in the box, thus checking your total.

Even experienced chemists seldom neglect this "check" method, because a single wrong weighing will spoil a whole experiment.

We shall conclude with a few further rules to be observed in weighing. The first two are specially important. (1) Unless for purposes of cleaning, adjustment, etc., *never remove a balance pan*. Once students begin doing this, pans are soon mixed and the balances are found to be hopelessly out of adjustment.

(2) Do not weigh chemicals on the bare scale pan.

(2) Do not weigh anything which is very hot. At least wait until you can comfortably bear your hand on it.

(4) Except for the heaviest weights (say the 100 gm. and the 50 gm.) use the forceps, not your fingers.

EXERCISE 4

METHODS OF MEASURING VOLUMES OF LIQUIDS

Object.—To study the apparatus and methods used in the laboratory for measuring the volumes of liquids.

Apparatus and Materials.—(a) *Beaker; stirring rod.* (b) *Measuring flask (say 250 c.c.); pipette (say 25 c.c.); graduate; burette; litmus solution; dilute sulphuric acid that has been further diluted to about 5 times its volume; sodium hydroxide solution similarly diluted.*

Directions, etc.—Notice the mark on the neck of the measuring flask. Probably "15°C" is also engraved, indicating that only at that temperature does the flask hold the recorded volume. For general class purposes it is sufficient if we avoid measuring a liquid when it is obviously warm.

The pipette is used for quickly measuring out small quantities of liquid. Fill it with water by suction, slightly

beyond the mark, and then close the end with the moistened forefinger of your right hand. Then let the surplus liquid escape till the bottom of the meniscus (*i.e.* the curved liquid surface) is opposite the mark.

Now let the 25 c.c. of water run into the beaker. Allow it to drain for about 15 seconds, the tip of the pipette touching the beaker. A small quantity of liquid remains in the pipette. *Do not blow this out*,—allowance has been made for it when constructing the instrument.

There is little to be said about the graduate except that in reading it, you must have your eye at the level of the liquid surface, and you read to the bottom of the meniscus.

These remarks apply also in reading the burette. It is a good plan, too, when reading it, to hold an envelope or piece of white cardboard behind it. The meniscus is then very clearly seen.

As an exercise in using the burette, fill it up with the sulphuric acid provided, and run some out until the acid is at (or a little below) the mark "0". Take the reading. Now add a few drops of litmus solution to the 25 c.c. of caustic soda in the evaporating dish, and slowly run in the sulphuric acid, controlling the burette tap with your right hand and constantly stirring with your left. When after stirring the colour of the liquid is just on the turn from blue to red, take the reading again, and subtract the first reading from it.

Repeat the work and compare your two results. They are good if they agree within 0.1 c.c. but you are not very likely to manage this at first.

It is important to manage the burette tap properly.

If it does not turn easily, rub on it a *very* small amount of vaseline (be careful not to block up the hole). As you turn the tap, press slightly inwards.

EXERCISE 5*

LAW OF MULTIPLE PROPORTIONS

Object.—To show that copper and chlorine combine in accordance with the law of Multiple Proportions.

Apparatus and Materials.—(a) *Bunsen; test-tube; boiling tube 6"×1"; beaker; hydrochloric acid; nitric acid.*
(b) *Balance and weights; thin copper foil (about 2½ gm.).*

Directions, etc.—The general principle of the experiment is as follows: A weighed quantity of copper (say x gm.) is converted, *via* the nitrate, into the oxide. The latter is dissolved in hydrochloric acid, forming cupric chloride, CuCl_2 . Thus we know the weight of copper (x gm.) which has combined with " Cl_2 ".

We now add a further weighed quantity of copper (y gm.)—more than enough to reduce the cupric chloride (CuCl_2) to cuprous chloride (Cu_2Cl_2). After the action is complete, we weigh the *excess* of copper (z gm.). Then $(y-z)$ gm. has reacted with the cupric chloride. The total weight of copper in the second chloride is therefore $x + (y-z)$.

Thus in the first case a certain weight of chlorine (" Cl_2 ") has combined with x gm. of copper, and in the second case with $x + (y-z)$ gm. We have to find by experiment if the ratio $x + (y-z) : x$ is a simple one.

As accurately as possible weigh two pieces of copper foil, one about 0.75 gm., the other about 1.5 gm., and put the smaller piece in the boiling tube. This piece may with advantage be first cut into thin strips, but this is not essential.

In a test-tube put about 1 c.c. of water and to this add about 4 c.c. of nitric acid. Add this mixture rather slowly to the copper in the boiling tube. *Q.1.* Complete the equation $\text{Cu} + 4\text{HNO}_3 \rightarrow$

As soon as the copper has completely dissolved, proceed to evaporate to dryness. In doing this, use a band of paper as a holder, and shake the boiling-tube rapidly from side to side. When dry, continue to heat strongly until the nitrate is completely decomposed into oxide. If there are any green splashes on the side, see that these are heated too. *Q.2.* What gas can you see coming off at this stage?

Test with a glowing splint. *Q.3.* Result?

Q.4. Write the equation representing the decomposition of the nitrate.

When the tube is cool enough to bear your hand on, add about 20 c.c. of hydrochloric acid, and heat till the copper oxide is completely dissolved. **Q.5.** Equation?

Now add the other piece of copper, and continue to boil gently till the green colour has completely disappeared, the cupric chloride, CuCl_2 having been reduced to cuprous chloride, Cu_2Cl_2 . **Q.6.** Equation?

Pour the contents of the tube into a beaker of water. The cuprous chloride is at once thrown down as a white precipitate.

Pick out the undissolved copper, wash it under the tap, and dry it *gently* (avoiding oxidation) by holding it about a foot above the top of a bunsen flame. Then weigh it.

Q.7. The results may be set out thus:—

Wt. of small piece of copper		x gm.
Wt. of larger piece		y gm.
Wt. of undissolved portion		z gm.
∴ Wt. of copper in 1st chloride	=	x gm.
and Wt. of copper in 2nd chloride		
(combined with the same wt. of chlorine)	=	$x + y - z$ gm.
∴ Ratio	=	$\frac{x + y - z}{x}$
	=	etc.

EXERCISE 6

REVERSIBLE REACTIONS

Object.—To illustrate the subject of reversible reactions and chemical equilibrium.

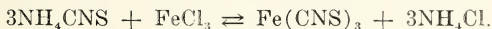
Apparatus and Materials.—(a) *Balances, etc.; beaker; stirring rod; test-tube; hydrochloric acid and hydrochloric acid dilute.* (b) *Four 8" gas-jars (2" int. diam.); graduate (100 c.c.); gummed paper (small piece); bismuth trichloride (2 gm.); solution of cryst. ferric chloride,*

$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, (8 gm. per litre), about 70 c.c.; solution of ammonium thiocyanate (12 gm. per litre), about 70 c.c.

Directions, etc.—1. Bismuth trichloride and water. Weigh out 2 gm. of bismuth trichloride and put it in a small beaker. Now add to it dilute hydrochloric acid (stirring all the time) until it *just* dissolves. Next add water gradually (still stirring) until a permanent white precipitate is obtained. Do not use more water than necessary. The change is represented by the equation $\text{BiCl}_3 + \text{H}_2\text{O} \rightleftharpoons \text{BiOCl} + 2\text{HCl}$. The white precipitate is bismuth oxychloride, BiOCl .

Again add hydrochloric acid (concentrated this time) till the precipitate *barely* redissolves. Then add water. *Q.1.* Can you once again obtain a precipitate? *Q.2.* Explain the fact that the addition of water causes a precipitate to be produced, while addition of hydrochloric acid causes the precipitate to be dissolved.

2. Ammonium thiocyanate and ferric chloride. This experiment is based on the reaction between ammonium thiocyanate (NH_4CNS) and ferric chloride (FeCl_3), producing ammonium chloride (NH_4Cl) and ferric thiocyanate ($\text{Fe}(\text{CNS})_3$); the latter having an intensely red colour.



Into each of four gas-jars, *P, Q, R, S*, pour 250 c.c. of water from the graduate. Pour 10 c.c. of water from a graduate into a test-tube, mark the level with gummed paper, and then use this marked test-tube to add 10 c.c. of the given ferric chloride solution to the water in each of the gas-jars. Rinse out the test-tube and then use it to add successive quantities of 10 c.c. of the given ammonium thiocyanate in the same way. Stir all four solutions. They will all have the same rather pale golden brown colour.

Keep *P* as a colour standard, and
(i) to *Q* add more ferric chloride solution (30 c.c. or so),

(ii) to R add more ammonium thiocyanate solution, and (iii) to S add about 30 c.c. of the given ammonium chloride solution. Q.3. What happens in each case? Q.4. Explain.

EXERCISE 7*

RAOULT'S LAW

Object.—To illustrate Raoult's Law. *If in equal quantities of a solvent we dissolve different substances (other than acids, bases, and salts) in quantities proportional to their molecular weights, the solutions thus obtained will all have the same freezing point.*

Apparatus and Materials.—(a) Three beakers; stirring rod. (b) Large evaporating dish (say 4" diam.); burette with stand¹; graduate (100 c.c.); thermometer reading -10° to 50°C. by 0.2° ; ice and salt for freezing mixture; urea (about 6 gm.); pure sodium chloride (about 6 gm.); absolute alcohol (about 6 c.c.).¹

Directions, etc.—First prepare solutions of urea, sodium chloride and alcohol as follows.

Into each of three beakers put 100 c.c. of water. Now counterpoise two small pieces of paper, weigh out 6 gm. of urea, and dissolve this in the first beaker. Similarly, dissolve 5.85 gm. of sodium chloride in the second.

Note that 6 is one-tenth of the molecular weight of urea, $\text{CO}(\text{NH}_2)_2$, and 5.85 is one-tenth of the molecular weight of sodium chloride.

The molecular weight of alcohol, $\text{C}_2\text{H}_5\text{OH}$, is 46, so we must add 4.6 gm. of alcohol to the third beaker. 4.6 gm. of alcohol = 5.8 c.c., and the simplest plan will be to run out this volume from a burette¹. All the solutions should be well stirred.

Find the freezing point of the first solution as follows. Fill the evaporating dish with a mixture of well-crushed

¹ It is convenient to make one burette, filled with alcohol, serve for a number of students.

ice (2 parts) and salt (1 part). Put about 5 c.c. of the solution into a test-tube, and introduce the latter in a nearly horizontal position as shown in Fig. 9, placing it so that you can just see the surface of the solution. The thermometer bulb should reach practically to the bottom of the test-tube, and by gently rattling it from side to side the liquid is kept in constant movement.

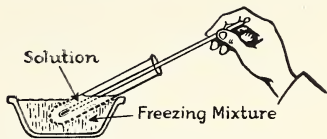


FIG. 9.

“Suspended solidification” is often noticed, *i.e.*, the temperature falls below the true freezing point, after which ice is seen to form and the mercury rapidly rises. As soon as the liquid is seen to be freezing, whether there has been suspended solidification or not, remove the test-tube, place it in a vertical position, and read the temperature as accurately as possible. Find the freezing point of the other solutions in the same way and (Q.1.) record your results.

Q.2. Account for the result obtained in the case of common salt.

EXERCISE 8

IONIZATION

Object.—To illustrate ionic equilibrium and its displacement.

Apparatus and Materials.—(a) Two test-tubes; hydrochloric acid. (b) Cupric chloride (a few gm.).

For additional experiments.—Cupric bromide, potassium bromide and sodium chloride, a few gm. of each.

Directions, etc.—*Experiment with cupric chloride.* Make a small quantity of a strong solution of cupric chloride in water. Q.1. What colour is it?

Add water little by little (shaking after each addition) until you can just detect a definite colour change. Q.2. What change do you notice?

Now add a little hydrochloric acid. Q.3. What further change (if any) do you notice? Q.4. Account for the changes if you can.

Additional Experiments

Experiment with cupric bromide. Put about 1 gm. of cupric bromide in a test-tube and dissolve it in the least possible quantity of water. Q.5. What colour is the solution? Add water gradually. Q.6. What colour changes do you notice? Q.7. Explain them. (*Hint.*—Brown may be regarded as a modified yellow.)

To the diluted solution gradually add a concentrated solution of potassium bromide. Q.8. Record and account for any other changes noticed.

Experiment with sodium chloride. Make a *strong* solution of sodium chloride in water, in a test-tube, and then add about an equal volume of hydrochloric acid. Q.9 What happens? Q.10. Can you account for the result?

EXERCISE 9

HYDROLYSIS

Object.—To show that when normal salts are dissolved in water, they give solutions which are sometimes acid, sometimes alkaline and sometimes neutral.

Apparatus and Materials.—(a) *Seven test-tubes; glass rod.* (b) *Copper sulphate, ferric chloride, sodium carbonate, sodium chloride, sodium borate (borax), potassium nitrate and aluminium sulphate (1 or 2 gm. of each); pieces of neutral litmus paper.*

Directions, etc.—Make a small quantity of copper sulphate solution in a test-tube. Take a drop of this on the end of a glass rod and touch a piece of neutral litmus paper with it. Q.1. Is the solution acid, alkaline, or neutral?

Repeat the experiment, using the following substances successively instead of copper sulphate:—ferric chloride, sodium carbonate, sodium chloride, sodium borate, potassium nitrate, and aluminium sulphate. *Q.2.* Complete the following table:—

<i>Salt</i>	<i>Nature of base and acid¹</i>	<i>Whether acid, alkaline, or neutral to litmus</i>
Copper sulphate	Wb + Sa	
Ferric chloride	Wb + Sa	
Sodium carbonate	Sb + Wa	
Sodium chloride	Sb + Sa	
Sodium borate	Sb + Wa	
Potassium nitrate	Sb + Sa	
Aluminium sulphate	Wb + Sa	

Q.3. Under what circumstances is a normal salt likely to give a solution which is (i) acid, (ii) alkaline, (iii) neutral to litmus?

EXERCISE 10

NEUTRALIZATION — I

Object.—To find the strength of a given specimen of dilute hydrochloric acid².

A normal solution of sodium hydroxide is provided.

Apparatus and Materials.—(a) *Retort stand; collar and clamp; beaker; stirring rod; test-tube.* (b) *Burette; 25 c.c. pipette; 250 c.c. measuring flask; glass tube, 8"*

¹ We may regard copper sulphate as being produced by the action of a strong acid (H_2SO_4) on a weak base ($\text{Cu}(\text{OH})_2$) — $\text{Cu}(\text{OH})_2 + \text{H}_2\text{SO}_4 \rightarrow \text{CuSO}_4 + 2\text{H}_2\text{O}$. Hence it is represented in the second column as Wb + Sa (weak base + strong acid). Similarly, sodium carbonate is represented as Sb + Wa (strong base + weak acid), and so on.

² *Note to teacher.* Say between 0.7N and 1.3N. See footnote 1, p. 18.

long; normal solution of sodium hydroxide¹, and the given specimen of hydrochloric acid, say, 100 c.c. of each; litmus solution, say 5 c.c.

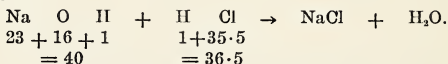
Preliminary Discussion.—Before beginning the experiment, it will be necessary to spend a little time in considering underlying theory.

Hydrochloric acid, HCl, has a molecular weight of $1 + 35.5 = 36.5$. Evidently 36.5 gm. of hydrochloric acid contains 1 gm. of hydrogen, and so 36.5 is known as the *equivalent* of hydrochloric acid.

The equivalent is not always the same as the molecular weight. For instance, the molecular weight of sulphuric acid, H_2SO_4 , is $2 + 32 + 64 = 98$. Now 98 gm. of sulphuric acid contains two gm. of hydrogen, and so the equivalent is 49—the equivalent being that weight of acid which contains 1 gm. of hydrogen.²

A *normal solution* is one of such strength that 1 litre of it contains the equivalent weight in grams. Thus a normal solution of hydrochloric acid contains 36.5 gm. of the pure acid in 1 litre. A normal solution of sulphuric acid would contain 49 gm. per litre.

Sodium hydroxide is neutralized by hydrochloric acid according to the equation



Now as 36.5 is the equivalent of hydrochloric acid, and it is neutralized by 40 of sodium hydroxide, we say that 40 is the equivalent of sodium hydroxide. A normal solution of sodium hydroxide would, therefore, contain 40 gm. per litre.

Since 36.5 gm. of hydrochloric acid neutralizes 40 gm. of sodium hydroxide, it is clear that 1 litre of a normal solution of hydrochloric acid (containing 36.5 gm.), will neutralize 1 litre of a normal solution of sodium hydroxide (containing 40 gm.), and, therefore, *any volume* of normal hydrochloric acid will neutralize *an equal volume* of normal sodium hydroxide. This principle, that *normal solutions are equivalent to one another, volume for volume*, is a very important one to grasp.

¹ Note to teacher. "Hydrochloric acid pure", diluted to 10 times its volume, gives a solution which is very nearly normal. A solution of sodium hydroxide of strength about 45 gm. per litre may be prepared, and then reduced to normality by the help of this hydrochloric acid (cf. Ex. 11).

² Strictly speaking, 1 gm. of hydrogen *replaceable by a metal*. Thus acetic acid, $\text{C}_2\text{H}_4\text{O}_2$, has a mol. wt. of $24 + 4 + 32 = 60$. Of the 4 gm. of hydrogen present in 60 gm. of acetic acid, only 1 gm. is replaceable by a metal (sodium acetate, for instance, is $\text{NaC}_2\text{H}_3\text{O}_2$). Hence the equivalent of acetic acid is 60, not 15.

Looking back to our "Object", suppose we were to find that 21.6 c.c. of the hydrochloric acid neutralizes 25 c.c. of *normal* sodium hydroxide.

Now if the hydrochloric acid had been normal, 25 c.c. of it would have been required ("normal solutions are equivalent to one another, volume for volume").

Clearly then, the hydrochloric acid is *stronger* than normal in the ratio of 25 to 21.6.

But a normal solution of hydrochloric acid contains 36.5 gm. per litre.

$$\therefore \text{This solution contains } \frac{25}{21.6} \times 36.5 \\ = 42.2 \text{ gm. per litre.}$$

It is evident that we can find the strength of the hydrochloric acid if we can find what volume of it is required to neutralize a given volume of normal sodium hydroxide; so we are now in a position to consider practical directions.

Directions, etc.—Wash out a burette and then rinse it with a little of the acid solution. *Q.1.* Why? Then fill it (or nearly fill it) with this solution. Run out a little so as to fill the part below the tap. *Q.2.* Why?

By means of a pipette, run 25 c.c. of the sodium hydroxide solution into a beaker. Close the top of the pipette with your right fore-finger—not your thumb—and when you let the solution flow into the beaker do not blow out the last drop. The pipette is constructed to deliver 25 c.c. by simply letting it drain for a quarter of a minute or so. (*Caution.* Be careful not to get sodium hydroxide solution into your mouth. If this *should* happen, do not swallow it. Spit it out and rinse your mouth with water.)

Now add two or three drops of litmus solution to the sodium hydroxide, to serve as indicator. A neat way of doing this is to have the litmus solution in a test-tube, and to pick it up with a piece of glass tubing as shown in Fig. 10.

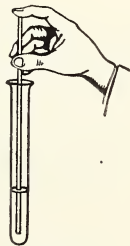


FIG. 10.

Now take the reading of the burette, and slowly run the acid into the beaker, stirring with your left hand and controlling the tap with your right. The beaker should stand on a sheet of white paper. Remember that the tap must all the time be pressed gently inwards, or the acid may flood out. When the litmus is just on the turn from blue to red, take the reading again. For hints on burette reading, refer to p. 10.

Repeat the "titration" (*i.e.* the process of measuring from a burette the amount of one solution required to react with another). The results are good if they do not differ by more than 0.1 c.c., but at first you are hardly likely to get as near as that. They may be set out something like this:—

	1st titration	2nd titration
Burette reading (<i>a</i>)	0.3	1.7
Burette reading (<i>b</i>)	31.6	33.1
Vol. of acid required (c.c.)	31.3	31.4
Mean result	31.35.	

The result is now worked out as already explained, but note that with the figures just given the acid would be *weaker* than normal ($25 \div 31.35$ of normal). *Q.3.* See that all numerical results are recorded, and that your method of calculation is clearly shown.

*Additional Exercise**

Find the strength of the ordinary "sulphuric acid dilute" used in the laboratory.

This acid will be much stronger than normal — four or five times perhaps. By a rough preliminary experiment get some idea of its strength. *E. g.*, if only 5.3 c.c. of the acid were required to neutralize 25 c.c. of normal sodium hydroxide, the acid is roughly 5 times normal ("5N").

In this case dilute the acid to exactly five times its volume. Find the strength of the *diluted* acid by the method already described, and multiply your answer by 5.

Two solutions should not be titrated against one another if there is a very great disparity in their strengths. Suppose we put 25 c.c. of a solution *A* in a beaker, and run *B* into it from a burette. In terms of normality, suppose *B* is only one-fifth as strong as *A*. We should require 125 c.c. of *B*, and the burette would have to be filled several times. On the other hand, if *B* were 5 times as strong as *A*, only 5.0 c.c. of it would be required, and in such a small quantity a slight experimental error would be serious.

Questions

1. Calculate the equivalent of (i) nitric acid, HNO_3 , (ii) phosphoric acid, H_3PO_4 . (N.B.—All three hydrogen atoms are replaceable by a metal). *Ans.* (i) 63. (ii) 32.7.
2. The dilute sulphuric acid used in chemical laboratories is frequently "5N", i.e. 5 times normal strength. The same applies to sodium hydroxide solution.
Calculate the weight of (i) sulphuric acid, (ii) sodium hydroxide present in 100 c.c. of each of these solutions. *Ans.* (i) 24.5 gm. (ii) 20 gm.
3. What volume of "5N" sulphuric acid would be required to neutralize 120 c.c. of normal potassium hydroxide solution? *Ans.* 24 c.c.
4. If you had 50 c.c. of a 0.39 normal solution of sodium hydroxide, what volume of 0.26 normal nitric acid would be required to neutralize it? *Ans.* 75 c.c.
5. Dilute sulphuric acid from a bench bottle is diluted to 5 times its volume, and it is found that 25 c.c. of the solution thus produced is neutralized by 20 c.c. of a potassium hydroxide solution of strength 63 gm. per litre. Find the strength (in gm. per litre) of the *original* sulphuric acid. *Ans.* 220.5.
6. A boy prepared a crude specimen of potassium carbonate from wood-ash (Ex. 30), and found that 1.25 gm. of it, dissolved in water, was neutralized by 21.8 c.c. of N/2 hydrochloric acid. Find the percentage of potassium carbonate in his specimen. *Ans.* 60.2.
7. 20 gm. of the acid from a storage battery is dissolved in water and the solution is made up to 250 c.c. 25 c.c. of the latter is neutralized by 27.2 c.c. of N/2 caustic soda solution. Find the percentage of sulphuric acid in the battery acid. *Ans.* 33.3.

EXERCISE 11*

NEUTRALIZATION—II

Object.—Given a normal solution of hydrochloric acid, and a solution of sodium hydroxide which is stronger than normal, to reduce the latter to normal strength.

Apparatus and Materials.—(a) *Beaker; retort stand; collar and clamp; stirring rod; test-tube.* (b) *Burette; glass tube about 8" long; 25 c.c. pipette; 100 c.c. measuring flask; graduate; normal solution of hydrochloric acid (say 250 c.c.)¹; solution of sodium hydroxide, about 50 gm. per litre (say 500 c.c.); litmus solution (say 5 c.c.).*

For Additional Exercise.—Sulphuric acid solution stronger than normal².

Directions, etc.—Proceed as before, putting the acid in the burette and 25 c.c. of the sodium hydroxide solution in the beaker. (As far as possible we avoid putting alkaline solutions in burettes, partly because the glass taps are very apt to become set fast.)

Since the sodium hydroxide solution is stronger than normal, it is evident that to neutralize 25 c.c. of it, more than 25 c.c. of normal hydrochloric acid will be required. Suppose the amount actually used is 29.1 c.c.

Now 29.1 c.c. of normal hydrochloric acid would neutralize 29.1 c.c. of normal sodium hydroxide. Therefore the solid sodium hydroxide present (in the 25 c.c. of "stronger than normal" solution which we used) is enough to make 29.1 c.c. of normal solution. Hence, to make our "stronger than normal" solution into a normal one, all we need do is to add 4.1 c.c. of water to every 25 c.c. of solution.

Measure the volume of the unused sodium hydroxide solution, using the measuring flask for the complete 100's, and the graduate only for the odd amount (the flask is

¹ *Note to teacher.* See Note 1 on p. 18.

² *Note to teacher.* The ordinary "dilute sulphuric acid" (5N), further diluted to 3 or 4 times its volume.

a more accurate measuring instrument than the graduate. Suppose the volume is 412 c.c. Then, amount of water to be added $= \frac{4.1}{25} \times 412 = 67.6$ c.c.

Add this amount of water, shake up *well*, and then do one more titration, this time using 25 c.c. of the diluted sodium hydroxide solution. Your work is very good if the amount of hydrochloric acid required is within 0.1 c.c. of 25 c.c. *Q.1.* See that all numerical results are recorded, and that your method of calculation is clearly shown.

*Additional Exercise**

Given a solution of sulphuric acid which is stronger than normal, use the normal sodium hydroxide solution which you have just made to prepare a normal solution of the acid.

Questions

1. Suppose you had 750 c.c. of dilute nitric acid, of such strength that 25 c.c. of it was neutralized by 30 c.c. of normal sodium hydroxide solution. How much water would you add to it in order to reduce it to normal strength? *Ans.* 150 c.c.
2. Calculate the weight of nitric acid present in 100 c.c. of the solution just mentioned (i) before, (ii) after diluting it with water. *Ans.* (i) 7.56 gm. (ii) 6.30 gm.

EXERCISE 12*

NEUTRALIZATION—III

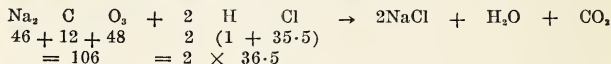
Object.—To find (i) the normality, (ii) the strength in gm. per litre, of the given solution of sodium carbonate, which contains between 20 and 30 gm. of sodium carbonate, Na_2CO_3 , per litre.

A semi-normal (N/2) solution of hydrochloric acid is provided.

Apparatus and Materials.—(a) *Beaker; retort-stand; collar and clamp; stirring rod; test-tube.* (b) *Burette;*

glass tube about 8" long; 25 c.c. pipette; semi-normal solution of hydrochloric acid, about 100 c.c. (cf. footnote 1, p. 18); about 100 c.c. of the sodium carbonate solution mentioned above (take between 54 and 81 gm. of sod. carb. crystals per litre); methyl orange solution, about 5 c.c.

Directions, etc.—First consider the equivalent of sodium carbonate. If we set down the equation—



we see that 106 gm. of sodium carbonate neutralizes 2×36.5 , i.e., two equivalents, of hydrochloric acid; and so 53 gm. would be required to neutralize *one* equivalent. Hence, equivalent of sodium carbonate = 53. Therefore, a normal solution would contain 53 gm. per litre, and a semi-normal solution 26.5 gm.

The given solution of sodium carbonate is, therefore, not very far out of semi-normal, and may conveniently be titrated against semi-normal acid. (It has already been explained on p. 21 that it is undesirable to titrate one solution against another if the two differ very greatly in respect of normality.)

The sodium carbonate solution is put in the beaker and the procedure is exactly as in previous exercises, except that methyl orange is used as indicator instead of litmus. To see why, go back to the equation. Our calculation will assume that a neutral solution is obtained when 73 parts (by weight) of hydrochloric acid have been added to 106 parts of sodium carbonate. Actually, however, owing to the presence of carbonic acid (from $\text{H}_2\text{O} + \text{CO}_2$), *less* than 73 parts of hydrochloric acid would be required. We must, therefore, either get rid of the carbonic acid by constant boiling, or what is more convenient, use an indicator (methyl orange) which is not affected by such a weak acid as carbonic.

Suppose the mean result is that 27.4 c.c. of N/2 acid = 25 c.c. of carbonate solution. If the carbonate solution had been N/2, only 25 c.c. of acid would have been required. But 27.4 c.c. was actually used.

\therefore the carbonate solution is stronger than N/2 in the ratio $27.4 : 25 = 1.096 : 1$.

∴ the carbonate solution is 1.096 of N/2, or 0.548 of normal, *i.e.*, *normality* = 0.548.

But, as already explained, a normal solution would contain 53 gm. per litre.

∴ this solution contains $53 \times 0.548 = 29.0$ gm. per litre.

Q.1. Record all burette readings, etc., and show how you arrive at your result.

Questions

1. It is found that to neutralize 25 c.c. of a solution of sodium hydroxide, 36 c.c. of N/2 sulphuric acid is required. Find (i) the normality of the hydroxide solution, (ii) its strength in gm. per litre. *Ans.* (i) 0.72. (ii) 28.8.
2. 4.08 gm. of "household ammonia" was diluted with water and neutralized with normal sulphuric acid, 25.2 c.c. of the latter being required. Find the percentage of ammonia, NH_3 , in the sample. *Ans.* 10.5.
3. 10.5 gm. of a certain base is dissolved in water and the solution is made up to 250 c.c. It is found that 20 c.c. of this is neutralized by 15 c.c. of normal sulphuric acid. Find the equivalent weight of the base. *Ans.* 56.0.
4. A chemist wishes to make up solutions of sulphuric acid and phosphoric acid of such strength that equal volumes of them shall be required to neutralize a given volume of potassium hydroxide solution. What relative weights of the acids will he take? *Ans.* 3 : 2.

EXERCISE 13

ELECTROCHEMICAL SERIES

Object.—To illustrate the fact that if a metal M_1 is above a metal M_2 in the electrochemical series,¹ then M_1 will displace M_2 from a solution of a salt of the latter.

Apparatus and Materials.—(a) Ten test-tubes on rack; dilute sulphuric acid. (b) About 15 c.c. of each of the following solutions:—lead nitrate, copper sulphate, silver nitrate, mercuric chloride, sodium chloride (5% strength in each case); gummed labels; 5 clean copper strips and 5 clean zinc strips each about 4" long by $\frac{1}{2}$ "; thread.

For Expt. 2—about 8" of magnesium ribbon.

¹ For this series, see your chemistry text-book.

Directions, etc.—1. (i) Take ten clean test-tubes, and half fill each of them with the following solutions:—
 (a) Two of them with lead nitrate, (b) two with dilute sulphuric acid¹ (c) two with copper sulphate, (d) two with silver nitrate, (e) two with mercuric chloride. Label them.

(ii) Into one of the test-tubes containing lead nitrate solution put a copper strip, and into the other a zinc strip. A piece of thread should be tied to each so that the strip can easily be withdrawn later. Deal similarly with the remaining four solutions.

(iii) In some cases there will be evidence of chemical change almost at once. Beginning with these, note any evidence for (or against) the statement made in the "Object."² Q.1. Notes may conveniently be set out in tabular form, thus:

	Lead nitrate	Sulphuric acid	Copper sulphate	Silver nitrate	Mercuric chloride
Copper strip					
Zinc strip					

2. Q.2. What would you expect to happen if you put some magnesium ribbon in solutions of sodium chloride, copper sulphate, and silver nitrate respectively? If time permits, find whether these results are actually obtained.

EXERCISE 14*

HYDROGEN FLUORIDE

Object.—To prepare hydrogen fluoride and to observe its action on glass.

¹ For the purpose of this exercise, hydrogen in sulphuric acid (hydrogen sulphate) may be regarded as a metal.

² Remember that a metal when deposited in a very finely divided condition is often black.

Apparatus and Materials.—(a) *Bunsen; test-tube; tripod stand; sulphuric acid.* (b) *Small lead dish; small sheet of glass (a cover-slip will do); pin; paraffin wax and calcium fluoride (a few gm. of each).*

Directions, etc.—Melt a little paraffin wax in a test-tube, and also warm a small sheet of glass. Pour the wax over the warm glass, moving the latter so that the wax shall flow all over it. Then stand the glass on its edge to drain and cool. This process should give a *thin* and fairly uniform coating.

Now with a pin scratch your initials, or some simple design, through the wax. Place the glass, wax side downwards, over a lead dish containing about 3 gm. of calcium fluoride which has been made into a paste with sulphuric acid. Place the dish on a tripod stand and warm it gently for a few minutes,—a quarter of an hour if possible,—taking care not to inhale the fumes. The latter attack the glass in the parts where it is not protected by wax, and, if the wax is scraped off, the design is found to be etched. *Q.1.* Write the equations representing the action of (i) sulphuric acid on calcium fluoride, (ii) hydrogen fluoride on silica.

EXERCISE 15

HYDROGEN BROMIDE AND BROMINE

Object.—To prepare hydrogen bromide and bromine, and examine their properties.

Apparatus and Materials.—(a) *Bunsen; 3 test-tubes and boiling tube; retort stand with ring; collar and clamp; retort; wire gauze; large beaker; glass rod; sulphuric acid.* (b) *Thistle funnel; potassium bromide (about 6 gm.); manganese dioxide (4 gm.); red calico, say 2" by 1¼"; 2 pieces of litmus paper (red and blue); chloroform (1 c.c.); silver nitrate solution; chlorine water.*

Directions, etc.—1. *Action of sulphuric acid on a bromide.* Remembering that hydrogen chloride is produced

by the action of sulphuric acid on a chloride, let us see if hydrogen *bromide* is produced by the action of sulphuric acid on a *bromide*.

Put about 1 gm. of potassium bromide in a test-tube, add a few drops of sulphuric acid (enough to cover it) and warm gently. *Q.1.* Are steamy fumes produced resembling hydrogen chloride? *Q.2.* What happens when you breathe across the mouth of the test-tube?

2. Action of hydrogen bromide on silver nitrate. Dip a glass rod in silver nitrate solution and hold a drop in the fumes. *Q.3.* What happens? *Q.4.* Write equations accounting for (i) the production of the steamy fumes, (ii) the change in the appearance of the silver nitrate.

Besides the steamy fumes of hydrogen bromide, you would be sure to notice the brown vapour of bromine. Sulphur dioxide is also produced, though it is often difficult to recognize the smell of this owing to the other gases present. *Q.5.* Regarding sulphuric acid as $\text{H}_2\text{O} \cdot \text{SO}_3$, suggest how this substance might react with HBr to give sulphur dioxide and bromine. *Q.6.* Complete the equation $\text{H}_2\text{SO}_4 + 2\text{HBr} \rightarrow$

3. Preparation and properties of bromine. When we wish to be sure that the hydrogen bromide shall be more completely converted into bromine, we use a special oxidizing agent, manganese dioxide.

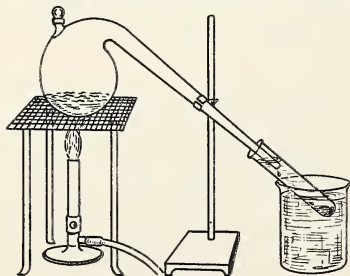


FIG. 11.

Into a test-tube pour 10 c.c. of water and cautiously add an equal volume of sulphuric acid. While the mixture is cooling, fit up the apparatus of Fig. 11, making the end of the retort dip just below the sur-

face of water contained in a wide test-tube. Into the retort put a well-ground mixture of potassium bromide (4 gm.) and manganese dioxide (4 gm.), taking care that none sticks on the sides. Using a thistle funnel, pour the diluted acid into the retort, quickly replace the stopper, and begin to heat gently. Bromine soon begins to collect at the bottom of the test-tube. *Q.7.* Note the colour and smell. *Q.8.* What can you say about its density and solubility?

Pour the *solution* of bromine into another test-tube. Fill up the first test-tube with water to about the same level as before, and shake up, so as to dissolve some more of the bromine from the bottom. Pour this solution also into another test-tube. We now have two test-tubes containing bromine solution, but free from undissolved bromine.

Into one of these, drop pieces of red and blue litmus paper, and a fragment of red calico. Leave them for a few minutes. *Q.9.* Are they bleached?

To the other add about 1 c.c. of chloroform and shake up. *Q.10.* What happens?

4. *Action of chlorine on a bromide.* Into another test-tube put two or three grains¹ of potassium bromide and dissolve in about 3 c.c. of water. Add a few c.c. of chlorine water. *Q.11.* What happens? *Q.12.* Write the equation.

EXERCISE 16

HYDROGEN IODIDE AND IODINE

Object.—To find whether hydrogen iodide can be obtained by a method similar to that used in preparing hydrogen chloride; also to prepare iodine and examine its properties.

Apparatus and Materials.—(a) *Bunsen; 4 test-tubes; beaker; tripod stand; wire gauze; evaporating dish; glass*

¹ This weight is fairly easy to judge roughly, for an average grain of dried wheat really weighs a grain.

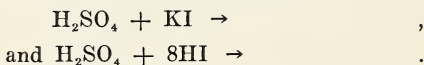
rod; sulphuric acid. (b) Potassium iodide (5 gm.); manganese dioxide (3 gm.); starch solution; alcohol (2 c.c.); chlorine water and bromine water (a few c.c. of each); ice (desirable but not essential).

Directions, etc.—1. *Action of sulphuric acid on an iodide.* On warming a chloride with sulphuric acid, hydrogen chloride is evolved. If we warm an *iodide* with sulphuric acid shall we obtain hydrogen *iodide*?

Try, using about a gram of potassium iodide in a test-tube, and a few drops of sulphuric acid. *Q.1.* What is the violet vapour?

Look carefully to see if you can make out any steamy fumes, and breathe across the mouth of the test-tube. *Q.2.* Does any gas resembling hydrogen chloride seem to be present?

Probably you can smell hydrogen sulphide (smell of bad eggs). *Q.3.* Can you suggest what has probably happened? *Q.4.* Complete the equations



The method just tried is evidently useless for the preparation of hydrogen iodide. This gas can be prepared by other methods, but we shall not consider them here.

2. *Preparation and properties of iodine.* We have seen that by the action of sulphuric acid on potassium iodide we obtain iodine. Better results are obtained if we add a special oxidizing agent (manganese dioxide).

Mix together about 3 gm. of powdered potassium iodide and 3 gm. of manganese dioxide. Put the mixture into a beaker and set the latter on a tripod stand (with wire gauze). Let an evaporating dish two-thirds full of cold water (if possible with a few pieces of ice floating in it) rest on the beaker.

Remove the evaporating dish for a minute while you add about 5 c.c. of sulphuric acid to the powdered mixture, stirring the powder into the acid with a glass rod. Replace

the evaporating dish and warm the beaker gently. Iodine is produced and sublimes on the under side of the dish and on the sides of the beaker.

Q.5. What colour is (i) the vapour, (ii) the solid? Has the solid any smell?

With the glass rod, scrape off a little of the iodine and transfer it to a test-tube half full of cold water. Shake up well for half a minute. *Q.6.* Is there any evidence that the iodine has dissolved?

Pour a few drops of starch solution into the test-tube. *Q.7.* What happens? (N.B.—The starch test for iodine is an extremely sensitive one. It works best with very *dilute* solutions of iodine.)

Into each of two other test-tubes put a few crystals of iodine. To one add about 2 c.c. of alcohol, to the other about 2 c.c. of potassium iodide solution. *Q.8.* Does iodine seem to be very soluble in these liquids? (N.B.—A solution of iodine in a mixture of these two liquids is known as *tincture of iodine*.)

To a little potassium iodide solution add a few drops of starch solution. *Q.9.* Does iodine give the blue colour when it is not in the free condition?

3. *Effect of chlorine and bromine on an iodide.* Put about 5 c.c. of potassium iodide solution into each of two test-tubes. To one add a few c.c. of chlorine water, to the other a little bromine water. *Q.10.* What seems to have happened? What makes you think so?

To each add a few drops of starch solution. *Q.11.* Was your suspicion correct? *Q.12.* Write equations representing the action of (i) chlorine, (ii) bromine on a solution of potassium iodide.

EXERCISE 17

CHLORIDES, BROMIDES, AND IODIDES

Object.—To learn how to distinguish between chlorides, bromides, and iodides.

Apparatus and Materials.—(a) *Bunsen*; 4 test-tubes; sulphuric acid; dilute nitric acid; ammonium hydroxide. (b) Potassium chloride, bromide and iodide (about 1 gm. of each); manganese dioxide (a few gm.); solution of silver nitrate; litmus paper; about 1 gm. of each of the substances A, B, and C (chloride, bromide, and iodide),—the student to find which is which.

Directions, etc.—1. *Behaviour with silver nitrate.*

(i) Dissolve a few grains of potassium chloride in about 2 c.c. of water in a test-tube, and add about 2 c.c. of silver nitrate. Q.1. What happens? Q.2. Write the equation.

Shake the precipitate so as to form a fairly uniform suspension, and then pour about half of it into another test-tube. To one portion add ammonia solution,—enough to half fill the test-tube,—and shake. Q.3. What happens?

To the other portion add dilute nitric acid,—also enough to half fill the test-tube,—and shake. Q.4. What happens?

(ii) Proceed as already described, but using potassium bromide instead of potassium chloride.

(iii) Proceed as before, but using potassium iodide. (N.B.—Silver chloride, bromide, and iodide are all insoluble in dilute nitric acid. This part of the test serves to distinguish them, not from one another, but from certain other precipitates, *e.g.*, silver phosphate, which are *soluble* in nitric acid.)

Q.5. Could you distinguish between silver chloride, bromide, and iodide by their colour? Q.6. Could you distinguish them by their solubility in ammonia?

2. *Behaviour with sulphuric acid and manganese dioxide.*

(i) Thoroughly mix about $\frac{1}{2}$ gm. of potassium chloride with rather more than that amount of manganese dioxide. Put the mixture in a test-tube, add enough sulphuric acid to soak it, and then warm gently. Hold a piece of damp litmus paper inside the mouth of the test-tube. Q.7. What happens to the litmus? Q.8. What gas is being evolved?

(ii) Repeat the test using potassium bromide and (iii) again, using the iodide; but in these two cases do not

use the litmus. Q.9. By the use of manganese dioxide and sulphuric acid, how can you distinguish between a chloride, bromide, and iodide?

3. Of the three salts A, B, and C, one is a chloride, one a bromide, and one an iodide. Find out which is which.

EXERCISE 18*

CALCIUM PHOSPHATE FROM BONES

Object.—To prepare calcium phosphate from bones.

Apparatus and Materials.—(a) Bunsen; small beaker; funnel; tripod stand; wire gauze; retort stand and ring; stirring rod; test-tube; hydrochloric acid and hydrochloric acid dilute; ammonia; silver nitrate. (b) Bone that has been boiled for a long time (e.g. in making soup)—about 5 gm.; iron mortar and pestle; graduate (100 c.c.); filter paper; balance, etc.; ammonium molybdate with nitric acid.

Directions, etc.—Take a small piece of the bone and break it up into small splinters in an iron mortar¹. Weigh out about 4 gm. of the product, and put it into a small beaker. If necessary wash several times by decantation, then add 20 c.c. of hydrochloric acid and heat nearly to boiling. In five minutes or so the bone should be entirely or almost entirely dissolved, but a little gelatine remains. To remove this add about 1 gm. of potassium chlorate. When the effervescence has subsided a little, heat to boiling, and continue to boil gently until no more chlorine is evolved.

Now dilute with water to 60 c.c., boil for a minute or two (to facilitate filtering) and filter.

The filtrate contains calcium phosphate, dissolved in the hydrochloric present. Add ammonia, stirring all the time, until the liquid smells of ammonia. Calcium phosphate will now be precipitated, because it is soluble only

¹ Note to teacher.—It saves much time if a supply sufficient for the class is broken up before the lesson.

in an acid solution. Boil once more, continue boiling for five minutes, and then filter.

The calcium phosphate left on the filter paper will contain various soluble impurities. (*Q.1.* Mention one or two.) If time permits it should, therefore, be washed with boiling water until the liquid filtering through no longer gives a precipitate with silver nitrate. (*Q.2.* Why this particular re-agent?)

The filter paper is now cautiously opened out, spread on a wire gauze, and placed some inches above a small bunsen flame to dry.

Test for a Phosphate. While drying, a little of the substance may be tested for a phosphate, as follows.

Dissolve by warming with dilute hydrochloric acid, and to *one or two drops* of the solution add ammonium molybdate solution and nitric acid. Warm. A fine yellow precipitate indicates a phosphate.

EXERCISE 19

ARSENIC AND SOME OF ITS COMPOUNDS

Object.—To study arsenic and some of its compounds.

Apparatus and Materials.—(a) *Bunsen*; 2 *test-tubes*; *glass rod*; *evaporating dish*; *sodium hydroxide solution*; *hydrochloric acid* and *hydrochloric acid dilute*. (b) *Flask fitted as in Fig. 13*; *Kipp's apparatus* generating *hydrogen sulphide* (alternatively, a strong solution of the gas); *litmus paper*; about 10 gm. *zinc* “free from arsenic, and antimony”; a few c.c. of *bleaching powder solution*.

Directions, etc.—1. *The Element.* Examine the stock-bottle of arsenic (the element). *Q.1.* Do you think it is a metal or a non-metal? Give your reasons.

2. *The Oxide.* Examine a little arsenious oxide (“white arsenic”. *Caution.* It is very poisonous.) Put a very little—about twice the volume of a grain of wheat—into a test-tube, add about 5 c.c. of water and boil. *Q.2.* Does it seem to dissolve?

Withdraw a drop of the liquid with a glass rod and put it on a piece of neutral litmus paper. *Q.3.* What happens? *Q.4.* Should you say that arsenious oxide is base-forming or acid-forming?

3. Shake up the turbid liquid just obtained and divide it into two parts. To one part add 2 or 3 c.c. of caustic soda solution and warm. *Q.5.* What happens? *Q.6.* Complete the equation $\text{As}_4\text{O}_6 + 12 \text{NaOH} \rightarrow$. (N.B.—Sodium arsenite is Na_3AsO_3 .)

To the other part of the turbid liquid add 2 or 3 c.c. of dilute hydrochloric acid and warm. *Q.7.* What happens? *Q.8.* Given that the substance formed in solution is arsenic trichloride, AsCl_3 , write the equation. Keep back a few drops of the solution just obtained, but pass hydrogen sulphide through the rest. *Q.9.* What happens? *Q.10.* Complete the equation.

4. *Reduction of the Oxide.* Into a clean dry test-tube (hard glass for preference) put (i) powdered charcoal to a depth of $\frac{1}{4}$ ", (ii) about 2 grains of arsenious oxide, (iii) about four times as much powdered charcoal as in (i). (The mixture is best introduced by putting it in the end of a long, narrow paper trough, which is then pushed to

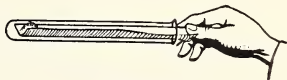


FIG. 12.

the bottom of the test-tube as indicated in Fig. 12. In this way the upper part of the tube is kept clean.) Now heat the upper part of the charcoal to bright redness, gradually working downwards. *Q.11.* What do you notice? *Q.12.* Write the equation representing the reduction of the oxide.

5. *Marsh's Test.* Fit up a flask with thistle funnel, etc., as shown in Fig. 13. In the flask put about 10 gm. of the granulated zinc provided, cover it with water and add hydrochloric acid. Collect a test-tube of the hydrogen evolved, light it at a bunsen at least a yard away, and then quickly bring the test-tube back to the jet. This is a safe

method. (*Caution.*—If hydrogen is lighted without some such precaution as this, a *dangerous explosion* may result.)

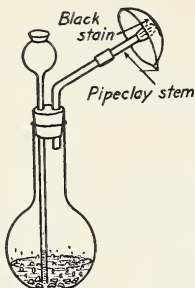


FIG. 13.

Add more acid from time to time as the evolution of gas slackens. Now hold a clean evaporating dish against the flame. If the zinc is pure there should be no black stain. Down the thistle funnel pour a few drops of the solution you kept back in Experiment 3. *Q.13.* What change do you notice in the flame?

Once more hold the porcelain dish in position. *Q.14.* What happens now?

Into the evaporating dish pour a little bleaching powder solution and shake gently. *Q.15.* Is the stain soluble?

EXERCISE 20

ANTIMONY AND SOME OF ITS COMPOUNDS

Object.—To study antimony and some of its compounds.

Apparatus and Materials.—(a) *Bunsen*; two test-tubes; beaker; stirring rod; hydrochloric acid; nitric acid. (b) Charcoal block; blowpipe; strip of zinc (say 4" by 2"); and (for general use) Kipp's apparatus and stock bottle of antimony.

For Expt. 6 only. Hydrogen generator with rt-angled delivery tube; evaporating dish; granulated zinc (free from Sb and As).

Directions, etc.—1. *The Metal.* Examine a quantity¹ of antimony. *Q.1.* Does it appear to be a metal or a non-metal? What makes you think so?

¹ A considerable quantity. The stock bottle might be passed around.

2. *Oxidation.* Heat a small quantity with a blowpipe on a charcoal block. In this case the charcoal serves merely as a support, and the element is oxidized to Sb_2O_3 .

Q.2. Where can you see this oxide? Write the equation.

3. *Chloride and Oxychloride.* To a very little of the metal add say 3 c.c. of hydrochloric acid and a few drops of nitric acid. On warming gently it dissolves, antimony chloride (SbCl_3) being formed. Pour the solution into 50 to 100 c.c. of water. The white precipitate formed is the oxychloride, SbOCl . Q.3. Write the equation.

Add hydrochloric acid drop by drop, stirring all the time, till the precipitate is just redissolved ($\text{SbOCl} + 2\text{HCl} \rightarrow \text{SbCl}_3 + \text{H}_2\text{O}$). Notice that the reaction proceeds from left to right (forming the soluble chloride) or vice versa (forming the insoluble oxychloride) according as we add *hydrochloric acid* or *water* (cf. bismuth chloride, Ex. 6.)

4. *Sulphide.* Pass hydrogen sulphide for a few seconds through some of the antimony chloride solution. Q.4. Describe the substance produced, and write the equation for the reaction which you think has taken place.

5. *Displacement.* Half fill a test-tube with some more of the antimony chloride solution and immerse a strip of zinc in it. Q.5. What happens, and how do you account for it?

6*. *Marsh's Test.* Repeat Expt. 5 of Exercise 19, but using a few c.c. of the antimony chloride solution instead of an arsenic compound. Q.6., Q.7. and Q.8. These questions correspond to Q.13., Q.14. and Q.15. in the last exercise.

EXERCISE 21

SODIUM HYDROXIDE

Object.—To prepare a solution of sodium hydroxide and to examine its properties.

Apparatus and Materials.—(a) *Bunsen*; two beakers; stirring rod; 4 test-tubes; tripod stand; wire gauze; dilute

hydrochloric acid. (b) *Pipette*; jar of carbon dioxide with greased cover; stick of caustic soda (2 or 3 ins.) in corked test-tube; ammonium chloride (a few gm.); 5 gm. washing soda; 5 gm. slaked lime; solutions of litmus, methyl orange and phenolphthalein (a few drops of each); solutions of lead nitrate, copper sulphate, ferric chloride and aluminium sulphate (a few c.c. of each).

Directions, etc.—1. Preparation. Dissolve 20 gm. of washing soda in 150 c.c. of water in a beaker, and add 10 gm. of slaked lime. Heat to boiling, and keep boiling for 3 minutes, stirring gently most of the time. The reaction is expressed by the equation $\text{Na}_2\text{CO}_3 + \text{Ca}(\text{OH})_2 \rightarrow \text{CaCO}_3 + 2\text{NaOH}$, and when it is complete there will be no carbonate in solution. Accordingly, after 3 minutes, let the insoluble material settle, and by means of a pipette transfer 1 or 2 c.c. to a test-tube. Add a little dilute hydrochloric acid. If there is no effervescence there is no carbonate present, and the reaction is complete. If there is effervescence the boiling must be continued a little longer.

Solid caustic soda could now be obtained by filtering the liquid in the beaker and boiling down, but it is hardly worth while to do this. If it is done, a funnel must be placed over the evaporating dish to prevent “spitting”, as with caustic soda this would be rather dangerous.

2. Appearance, Solubility, etc. Take the caustic soda stick from the corked test-tube. Notice how it “sweats” on exposure to the air, i.e., it is *deliquescent*. Break the stick into two or three pieces (protecting your fingers with paper) and put these into a beaker. Now add a little water and stir. Notice the great solubility of the substance and the large amount of heat produced.

For the remaining experiments you may use either this solution or the solution obtained in Expt.1.

3. Rub the solution between your fingers (afterwards rinsing well). *Q.1.* What do you notice?

4. *Effect on Indicators.* Put a little solution into each of three test-tubes, and add a few drops of litmus to the first, of methyl orange to the second, and of phenolphthalein to the third. Q.2. What happens?

5. *Effect on Acids.* We have already seen (Ex. 10) that caustic soda neutralizes acids. The substances produced are *salts*.

6. *Effect on Carbon Dioxide.* Remove the cover-slip from the jar of carbon dioxide, pour in caustic soda solution to a depth of about an inch and quickly replace the cover, "screwing" the latter well down. Shake up, invert the jar in a trough or sink of water, and remove the cover-slip. (This may be difficult. A side-ways movement is best.) Q.3. What happens? Q.4. Write the equation for the action of caustic soda on carbon dioxide.

7. *Production of Hydroxides.* Into four different test-tubes put small quantities of the following solutions:—lead nitrate, copper sulphate, ferric chloride, and aluminium sulphate. To each add a little caustic soda solution. This causes the respective *hydroxides* to be precipitated. Q.5. Write the equations.

8. *Reaction with Ammonium Salts.* Put a pinch of ammonium chloride in a test-tube, add caustic soda solution, and warm gently. Q.6. What can you smell? Q.7. Equation? (N.B. Other ammonium salts react in a similar way.)

EXERCISE 22

OTHER COMPOUNDS OF SODIUM

Object.—To study some other compounds of sodium.

Apparatus and Materials.—(a) *Bunsen burner* (*Meker* if available); 3 test-tubes; retort stand; collar and clamp; hydrochloric acid dilute; lime-water. (b) *Hard glass test-tube fitted with rubber stopper and delivery tube* (Fig. 14); another hard glass tube; pneumatic trough; platinum wire

in holder¹; wooden splint; sodium nitrate and bicarbonate, about 10 gm. of each; sodium peroxide, about 1 gm.

Directions, etc.—1. *Effect of heat on sodium nitrate.* Fill a hard glass test-tube about a third full with sodium

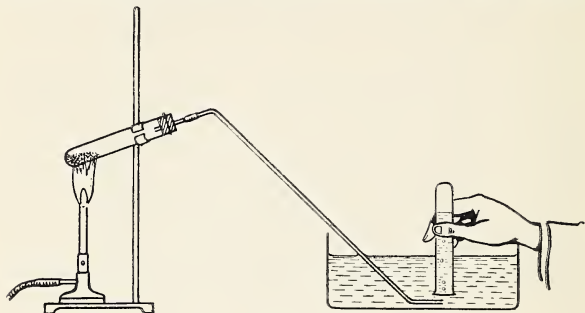


FIG. 14.

nitrate, fit it with a cork and delivery tube as shown in Fig. 14, and arrange for the collection of a gas over water. The test-tube in which the gas is to be collected may be held either with a clamp or by the hand.

After "warming up" the hard glass tube, heat it as strongly as possible—if a Meker burner is available so much the better. Give time for the air to be expelled from the tube, and then collect the gas given off. Test the latter with a glowing splint. *Q.1.* What happens, and what is the gas?

After letting the test-tube cool down, add dilute hydrochloric acid to the contents. Also add this acid to a little sodium nitrate. *Q.2.* What happens in the two cases? Is sodium nitrate changed by heating?

¹ Instead of platinum wire, a porcelain rod (something like that used for supporting an incandescent mantle) may be used. The rods may be bought fairly cheaply by the gross from dealers in chemical apparatus. Another useful substitute is the "lead" obtained by cutting open a lead pencil.

Actually the residue is sodium *nitrite*, NaNO_2 .
Q.3. Write the equation representing the decomposition of sodium nitrate.

2. Effect of water on sodium peroxide. Change the test-tube just used for a clean one (which need not, however, be of hard glass). The apparatus is arranged as before, except that no bunsen will be needed. Place about a gram of sodium peroxide at the bottom of the test-tube (Fig. 12, p. 35), add a few c.c. of water and quickly replace the cork. After allowing a little interval for air to be displaced, collect a test-tube of the gas, and test this also with a glowing splint. *Q.4.* What happens, and what is the gas?

Rub a few drops of the contents of the test-tube between your thumb and finger (afterwards rinsing under the tap).
Q.5. What does the liquid appear to be? *Q.6.* Write the equation for the action of water on sodium peroxide.

3. Effect of heat on sodium bicarbonate. Repeat Expt. 2, using sodium bicarbonate instead of nitrate. In this case an ordinary bunsen will give quite sufficient heat. When a test-tube of gas has been obtained add lime-water to it. *Q.7.* What happens, and what is the gas?

Notice the upper part of the test-tube, and the delivery tube. *Q.8.* Does any other substance (besides the gas) appear to have been produced in the course of the experiment?

Taste about a grain (foot-note, p. 29) of (i) sodium bicarbonate, (ii) the substance left in the test-tube. *Q.9.* Do you notice any difference? *Q.10.* If so, can you account for it? *Q.11.* Write the equation for the action of heat on sodium bicarbonate.

4. Flame Test for Sodium Compounds. This is described on p. 87.

EXERCISE 23*

ATOMIC WEIGHT OF SODIUM

Object.—Assuming that when sodium bicarbonate is heated it decomposes in accordance with the equation $2\text{NaHCO}_3 \rightarrow \text{Na}_2\text{CO}_3 + \text{H}_2\text{O} + \text{CO}_2$, and that we know

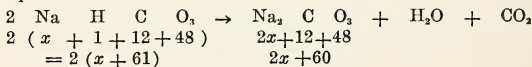
the atomic weights of the other elements concerned, to find the atomic weight of sodium.

Apparatus and Materials.—(a) *Bunsen; balance and weights; evaporating dish; tripod stand; wire gauze; crucible tongs.* (b) *Desiccator (if available); sodium bicarbonate (say 20 gm.).*

Directions, etc.—Weigh an evaporating dish, first empty and then about a quarter full of sodium bicarbonate. Put it on wire gauze on a tripod stand and heat it for 20 minutes. Allow it to cool—in a desiccator if one is available—and weigh again. If time permits, the dish should be heated for another 5 minutes, to make sure that constant weight has been attained, showing that chemical change is complete. The method of arriving at the atomic weight will be clear from the following example.

(a)	Wt. of empty dish	43.26 gm.
(b)	Dish + sod. bicarb.	54.67 gm.
(c ₁)	Dish + sod. carb. (after 1st heating)	50.47 gm.
(c ₂)	Dish + sod. carb. (after heating another 5 mins.) ¹	50.46 gm.
∴	Wt. of sod. bicarb. = (b) — (a)	= 11.41 gm.
	and wt. of sod. carb. = (c ₂) — (a)	= 7.20 gm.
∴ ratio	$\frac{\text{wt. of sod. bicarb.}}{\text{wt. of sod. carb.}}$	= $\frac{11.41}{7.20}$ (A).

Now let x be required atomic weight of sodium, and consider the equation.



$$\text{This gives } \frac{\text{wt. of sod. bicarb.}}{\text{wt. of sod. carb.}} = \frac{2(x+61)}{2x+60} \quad (B).$$

Equating (B) to (A) we have

$$\frac{2(x+61)}{2(x+30)} = \frac{11.41}{7.20}.$$

Solving, we get $x = 23.0$.

Q.1. Record all weighings, and show how you obtain your result, as in the example given.

¹ If (c₂) differed considerably from (c₁), further heating would be necessary.

EXERCISE 24*

FORMULA OF GLAUBER SALT

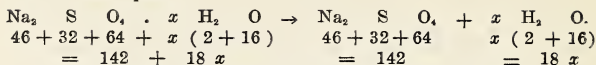
Object.—Given that Glauber salt has the formula $\text{Na}_2\text{SO}_4 \cdot x\text{H}_2\text{O}$, to find the value of x .

Apparatus and Materials.—(a) *Bunsen; tripod stand; pipeclay triangle; crucible tongs.* (b) *Balance, etc.; porcelain crucible with lid; Glauber salt, 4 or 5 gm. (It is important that this should have been kept in a well-stoppered bottle; the crystals should show no sign of efflorescence.)*

Directions, etc.—Weigh the crucible and lid, first empty and then about one-third full of the Glauber salt. Place the crucible on the pipeclay triangle, the latter resting on the tripod stand. The lid should cover the crucible except for a gap of about $\frac{1}{8}$ ". Heat cautiously—there is a tendency for small crystals to fly out of the crucible (*Q.1.*—Why?). It is best to heat for a second or two until steam is seen to be escaping pretty freely and then to withdraw the bunsen. After perhaps two minutes of this intermittent heating, steam no longer seems to be evolved. The lid is then drawn back a further $\frac{1}{4}$ " or so and the crucible heated strongly for another five minutes. It is then cooled in a desiccator (if one is available) and weighed, the lid being kept on throughout. To make sure that the chemical action is complete, the crucible should be strongly heated for another two minutes, cooled and weighed again. The final weight should, of course, be the same as the preceding one. *Q.2.* Enter your results and work them out as follows:

Wt. of crucible (with lid throughout)	12.25 gm.
Crucible + salt	14.88 gm.
do. after heating 1st time	13.41 gm.
do. after heating 2nd time	13.41 gm.
Wt. of Glauber salt = $14.88 - 12.25 =$	2.63 gm.
and wt. of water = $14.88 - 13.41 =$	1.47 gm.
2.63 gm. of Glauber salt loses	1.47 gm.
$\therefore 1 \text{ gm. of Glauber salt loses } \frac{2.63 \text{ gm.}}{1.47 \text{ gm.}} \text{ A.}$	

From the equation



We have $142 + 18x$ gm. Glauber salt loses $18x$.

$$\therefore 1 \text{ gm. Glauber salt loses } \frac{18x}{142 + 18x}. \quad (B).$$

Equating (A) and (B) we obtain the value of x .

Additional Exercise

Repeat the experiment with soda crystals.

Questions

The percentage of water of crystallization on certain hydrated salts is as follows:—Copper sulphate, 36.0; Epsom salt, $\text{MgSO}_4 \cdot x\text{H}_2\text{O}$, 51.2; ferrous sulphate, 45.3; alum, $\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$, 45.6; sodium phosphate, $\text{Na}_2\text{HPO}_4 \cdot x\text{H}_2\text{O}$, 60.3. In each case calculate the number of molecules of water of crystallization. *Ans.* 5; 7; 7; 24; 12.

EXERCISE 25

PROPERTIES OF SODIUM THIOSULPHATE

Object.—To examine the properties of sodium thiosulphate.

Apparatus and Materials.—(a) *Bunsen*; 3 test-tubes; dilute hydrochloric acid; silver nitrate solution; chlorine water (freshly made). (b) Sodium thiosulphate, sodium sulphite and potassium bromide, a few gm. of each; solution of iodine in potassium iodide (a few c.c.).

Directions, etc.—To a few crystals of the thiosulphate add dilute hydrochloric acid and warm gently. Smell.

Q.1. What gas is coming off? **Q.2.** Judging by the appearance of the liquid in the test-tube, what other substance is being produced? **Q.3.** Write the equation expressing the reaction.

Repeat the experiment (*i.e.*, adding dilute hydrochloric acid and warming) with a few crystals of sodium sulphite. *Q.4.* In what way is the result similar to that obtained in the last experiment, and in what way is it different? *Q.5.* Write the equation.

Dissolve a few grains of potassium bromide in water and add silver nitrate solution. Silver bromide is precipitated (*Q.6.* Equation?) Add "hypo" solution and shake up. *Q.7.* What happens? *Q.8.* Given that the soluble substance formed is sodium silver thiosulphate, NaAgS_2O_3 , write the equation.

Add "hypo" solution to a solution of iodine in potassium iodide. *Q.9.* What happens? *Q.10.* Given that one of the substances formed is sodium tetra thionate, $\text{Na}_2\text{S}_4\text{O}_6$, complete the equation $2\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 \rightarrow$

Now add "hypo" solution to a little chlorine water and shake up. *Q.11.* Can you smell chlorine now? *Q.12.* Write the equation.

EXERCISE 26*

PREPARATION OF POTASSIUM CHLORATE

Object.—To prepare potassium chlorate.

Apparatus and Materials.—(a) Bunsen; tripod stand; wire gauze; 20 c.c. beaker, of tall shape; hydrochloric acid.

(b) Flask, fitted as shown in sketch (but see "Directions"); thermometer (desirable, but not essential); balance, etc.; graduate; potassium permanganate (25 gm.); potassium hydroxide (30 gm.).

Directions, etc.—In a fume-cupboard fit up the apparatus shown in the figure for the preparation of chlorine. The flask contains 25 gm. of potassium permanganate, and about

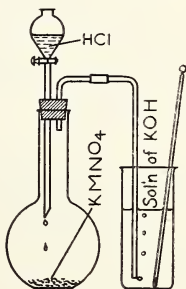


FIG. 15.

150 c.c of hydrochloric acid will have to be added through the dropping funnel. (If the latter is not available, use an ordinary thistle funnel, all but touching the bottom of the flask, and pour in a little water to cover the bottom of the funnel. The acid will then be added a little at a time.

Now dissolve 30 gm. of caustic potash in 80 c.c. of water in the beaker. The solution will be hot, but heat it a little more if necessary to bring it to 70°C . Keep a thermometer in the beaker and allow the acid to drip on the permanganate, the stream of chlorine passing through the solution. Keep the temperature near to 70°C . by allowing the acid to run in a little faster or slower as required.

After passing chlorine for 20 - 25 minutes the temperature suddenly rises and chlorine ceases to be absorbed. Cool the beaker under the tap. Crystals soon begin to be deposited, and after leaving for a few hours there will be a good crop.

Pour off the mother liquor, wash two or three times with a *little* cold water, and dry between blotting paper.

- Q.1.* Complete the equation $\text{KMnO}_4 + 8\text{HCl} \rightarrow$.
 (N.B.—“8H” has been taken, to combine with 4 atoms of oxygen. KCl and MnCl_2 will be formed.) *Q.2.* Complete the equation $6\text{KOH} + 3\text{Cl}_2 \rightarrow 5\text{KCl} +$.
Q.3. Make a labelled sketch of your apparatus.

During a subsequent visit to the laboratory, when you have got some dry crystals, it is worth while to heat a small quantity in a test-tube and try the glowing splint test for oxygen.

EXERCISE 27

DENSITY OF OXYGEN AND PERCENTAGE OF OXYGEN IN POTASSIUM CHLORATE

Object.—To find (i) the weight of a litre of oxygen at N.T.P. and (ii) the percentage of oxygen in potassium chlorate.

Apparatus and Materials.—(a) Bunsen; collar and clamp; retort stand. (b) Test-tube (hard glass if possible), fitted with rubber stopper through which passes a short glass tube; about 2 ft. of rubber tubing; flask (about 500 c.c.); trough; balance, etc.; graduate; barometer; thermometer; a few gm. of manganese dioxide, powdered, which just before the lesson has been heated for a few minutes in an open dish; powdered potassium chlorate (a few gm.).

Directions,
etc.—Weigh the test-tube (including stopper and short piece of glass tubing). Suppose weight = a . Introduce about 1.5 gm. of potassium chlorate and weigh again (b). Then introduce about 0.5 gm. of the manganese dioxide and weigh again (c). Evidently wt. of potassium chlorate = $b - a$.

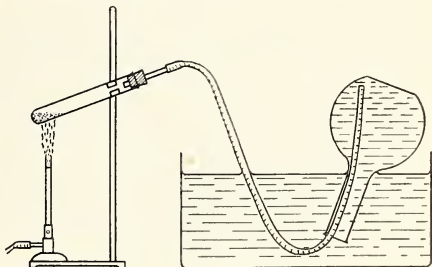


Fig. 16.

Shake well so as to mix up the potassium chlorate and the manganese dioxide. Attach the rubber tubing. Fill the flask with water and invert it in the trough, taking care that no bubbles of air enter, and make the rubber tubing pass well into the flask, say an inch or so from the top. Secure the test-tube by means of a clamp fixed near the cork (so as to be out of the way of the bunsen flame). Now heat the test-tube¹ until no more gas is evolved, and then let it cool to room-temperature, the end of the rubber tube being surrounded by the oxygen in the flask. As

¹ There should be at least a little space between the upper surface of the mixture and the test-tube, and heating should begin from the bottom end.

the test-tube cools the oxygen contracts, and water rises correspondingly in the flask.

When cooling is complete, push the flask down into the trough until the water-level is the same inside and out. Then, after detaching the rubber tubing from the glass tube, withdraw it altogether.

Cork the flask, find the volume of the contained water and the total volume of the flask. The difference is evidently the volume of the oxygen. Read the barometer, take the temperature of the water in the trough, and reduce the volume of oxygen to N.T.P. Suppose this corrected volume is v . Now weigh the tube with cork, etc. Suppose this wt. is d . Then the *weight* of oxygen driven off is $c - d$. The *volume* of the oxygen at N.T.P. is v .

$$\therefore \text{wt. of 1 litre at N.T.P.} = \frac{(c - d)1000}{v} \text{ gm.}$$

Also, percentage of oxygen in potassium chlorate

$$= \frac{c - d}{b - a} \times 100.$$

EXERCISE 28

POTASSIUM NITRATE

Object.—To prepare potassium nitrate and examine its properties.

Apparatus and Materials.—(a) *Bunsen; balance, etc.; boiling tube; sulphuric acid; hydrochloric acid dilute.* (b) *Extra boiling tube; graduate; hard glass test-tube fitted with rubber stopper and delivery tube (Fig. 13); pneumatic trough; splint; platinum wire in holder or substitute¹; blue glass; sodium nitrate, potassium chloride and potassium nitrate (about 15 gm. of each); copper powder or turnings (a pinch).*

Directions, etc.—1. *Preparation of potassium nitrate.* Weigh up roughly 13 gm. of sodium nitrate and 11 gm.

¹ See foot-note p. 40.

of potassium chloride, and add these to 25 c.c. of hot water contained in a boiling tube. Now boil the liquid. The substances will dissolve completely, but if "commercial" materials have been used there will be some undissolved sand, etc., which should be removed by filtration, the clean liquid filtering through into another boiling tube¹. Leave the latter for a few hours, when crystals of potassium nitrate will have separated out. *Q.1.* Write the equation.

2. Effect of heat. This experiment is carried out exactly as described under "Effect of heat on sodium nitrate" (p. 40). Answer the questions given there, but read "potassium" for "sodium", and number them *Q.2*, *Q.3*, *Q.4*, instead of *Q.1*, *Q.2*, *Q.3*.

3. Effect of sulphuric acid. Heat a little potassium nitrate with sulphuric acid. *Q.5.* Is there any sign of the production of nitric acid? (*Hint.*—Nitric acid on heating undergoes a certain amount of decomposition, brown fumes of nitrogen peroxide being produced. These fumes will be more readily visible if you look down into the test-tube, but do not hold it near your face.)

4. "Copper" test for a nitrate. Repeat the last experiment, but first mix a little copper with the potassium nitrate. *Q.6.* What difference do you notice? *Q.7.* Account for the difference.

5. Flame test. Try the flame test for potassium (Ex. 50). *Q.8.* Result?

¹ If at this stage the solution is transferred to an evaporating dish and boiled down to about two-thirds its volume, crystals will form much more quickly, and in larger quantity.

EXERCISE 29

PREPARATION OF POTASSIUM BROMIDE AND IODIDE¹

A. Potassium Bromide

Object.—To prepare a specimen of potassium bromide.

Apparatus and Materials.—(a) Bunsen (two, if possible); evaporating dish; tripod stand; wire gauze; two test-tubes; boiling tube; glass rod. (b) Potassium hydroxide (preferably in “pellet” form), 5.6 gm.; burette, in stand, containing bromine (see footnote 2); splint.

Directions, etc.—Counterpoise two pieces of paper, and weigh out 5.6 gm. of potassium hydroxide. Put this in a small evaporating dish and dissolve in 20 c.c. of water. Now add bromine slowly from a burette, taking care to press gently inwards on the tap. Stir the solution while the bromine is being added. At first, a very pale yellow colour is noticed, becoming gradually deeper. At a certain stage, this deepening sets in rather suddenly, indicating that bromine is now in excess. To make quite sure, add 0.2 c.c. more. For the quantity of hydroxide taken, the amount of bromine added should in no case exceed 3 c.c. *Q.1.* Write the equation.

Evaporate to dryness, heating cautiously so as to avoid loss by “spitting” as far as possible. *Q.2.* What colour changes do you notice as the evaporation proceeds? *Q.3.* What colour is the residue?

As soon as the product has ceased steaming transfer a little of it to a test-tube and heat (meanwhile, continue to heat the evaporating dish, with the bunsen full on).

¹ *Notes to teacher.* 1. Half the class might do the bromide, and the other half the iodide.

2. It is not advisable for the students to handle the stock bottle of bromine. It is suggested that the bromine be placed in the burettes for them, care being taken to see that the part below the tap is full. The burettes should be placed in fume cupboards if possible, and it is better to have a minimum number in use,—say one to every five students.

Q.4. Is any oxygen evolved from the substance in the test-tube (glowing splint test)? Q.5. How do you account for it?

After the substance in the evaporating dish has been heated for 10 minutes (*i.e.* from the time that it ceased steaming), repeat the test-tube experiment. Q.6. Result?

The heating of the evaporating dish must be continued if necessary until all bromate has been decomposed, but with a good bunsen the 10 minutes mentioned will be sufficient.

Let the dish cool, and then detach the contents with the point of a knife. To obtain fair-sized crystals, transfer the substance to a boiling tube, add 10 c.c. of water and boil for a minute. If necessary add a further 2 or 3 c.c., and boil again. Then leave the liquid to crystallize. Q.7. What shape are the crystals?

B. Potassium Iodide

Object.—To prepare a specimen of potassium iodide.

Apparatus and Materials.—(a) *Bunsen; evaporating dish; tripod stand, wire gauze; test-tube; pipeclay triangle; beaker; funnel.* (b) *Crucible (12 c.c. or larger); small pestle and mortar; splint; filter paper; potassium hydroxide (preferably in “pellet” form) 2.8 gm.¹; iodine, 5.4 gm.¹; powdered charcoal, 2 gm.*

Directions, etc.—Counterpoise two pieces of paper, and weigh out (i) 2.8 gm. of potassium hydroxide, and (ii) 5.4 gm. of iodine.

Dissolve the caustic potash in 10 c.c. of water in a test-tube, and add the iodine little by little, shaking after each addition, until the liquid begins to acquire a permanent brown colour. It is rather easy to overstep the mark. In that case a little more caustic potash solution must be added.

¹ It would really be better to double these quantities, but the cost of iodine is a consideration.

Pour into an evaporating dish and cautiously evaporate to dryness.¹ Put a *small* portion of the residue into a test-tube and heat. *Q.1* Is any oxygen evolved (glowing splint test)? *Q.2*. How do you account for it?

Grind up the remaining portion in a small mortar with 2 gm. of powdered charcoal. Transfer the mixture to a crucible, cover it with the lid, and then heat strongly on a pipeclap triangle for 15 minutes. This causes any iodate present to be reduced to iodide. *Q.3*. Equation?

The soluble iodide must now be separated from the insoluble charcoal. Accordingly, as soon as the crucible is cool enough to handle, place it (without the lid) in a beaker, cover it with water, boil, and filter into a clean evaporating dish. Now boil down *nearly* to dryness and allow to crystallize out. *Q.4*. Can you make out the shape of the crystals?

EXERCISE 30*

POTASSIUM CARBONATE FROM WOOD-ASH

Object.—To examine wood ash, and to prepare from it a crude specimen of potassium carbonate.

Apparatus and Materials.—(a) *Bunsen*; 2 *test-tubes*; *beaker* (250 c.c.); *stirring rod*; *evaporating dish*; *funnel and stand*; *tripod stand*; *wire gauze*; *crucible tongs*; *lime-water*; *dilute hydrochloric acid*. (b) *Wood-ash* (well burnt; about 30 gm.); *platinum wire in holder*²; *sheet of blue glass*; *filter paper*; *small crucible* (*nickel for preference*).

Directions, etc.—1. *Action of dilute hydrochloric acid*. Put a little wood-ash in a test-tube and pour dilute hydrochloric acid on it. *Q.1*. What happens?

¹ "Spitting" is usually very troublesome in this case. If time permits it is much better to use a water-bath.

² See foot-note p. 40.

Put some lime-water in a second test-tube, and hold the two test-tubes mouth to mouth as shown in Fig. 17, with the thumb passing over the top to help make a sort of tunnel. In this way a considerable amount of gas passes into the second test-tube, which after half a minute or so is shaken up.



FIG. 17.

Q.2. Is the gas carbon dioxide? *Q.3.* What sort of compound must have been present in the wood-ash?

2. Flame Test. Apply the flame test (Ex. 50) to the wood-ash, observing the flame through a sheet of blue glass. *Q.4.* What colour do you notice? (N.B. A violet colour—nearly crimson when observed through blue glass—indicates a *potassium* compound.)

3. Extraction of soluble matter. Boil 100 c.c. of water in a 250 c.c. beaker, and to the boiling water add 25 gm. of wood-ash. Continue to boil for another five minutes or so, when a good part of the insoluble material will settle. Filter into an evaporating dish, disturbing the sediment as little as possible.

Add 50 c.c. of water to the sediment, boil, allow to settle as before, and filter into the same evaporating dish. Place the latter on wire gauze and evaporate to dryness. The residue is very impure potassium carbonate, and often has an earthy colour. To remove some of the impurities, melt it in a crucible placed on a pipe-clay triangle, heating it till the molten substance becomes clear.

After cooling, grip the crucible with tongs and hold it sideways in a weighed evaporating dish half full of water, which is being heated. In a little while the contents of the crucible are dissolved out. Filter if necessary, and then gently evaporate to dryness. *Q.5.* What weight of potassium carbonate have you obtained?

Additional Exercise

Dissolve your specimen in about 25 c.c. of water, add methyl orange to serve as indicator, and then from a burette add a N/1 solution of hydrochloric acid till neutral. Calculate the percentage of potassium carbonate in the specimen.

EXERCISE 31*

HARDNESS OF WATER

Object.—To find the proportions of temporary and permanent hardness respectively in a specimen of water.

Apparatus and Materials.—(a) *Bunsen; beaker; tripod stand; wire gauze; lime-water; hydrochloric acid.* (b) *Flask containing marble chips, and fitted with thistle funnel, etc., for generating carbon dioxide*¹; 100 c.c. graduate; burette; 25 c.c. pipette; ordinary 6 oz.-medicine bottle, with cork; soap solution²; strong solution of sodium carbonate (10 c.c.); saturated solution of calcium sulphate (30 c.c.); distilled water (say 50 c.c.).

Directions, etc.—(a) *Preparation of specimen.* The water of the district may be suitable for this experiment (ask your teacher). If not, prepare a specimen artificially by adding 120 c.c. of distilled water to 50 c.c. of lime-water, and passing carbon dioxide until the calcium carbonate first precipitated is redissolved. Then add 30 c.c. of (saturated) calcium sulphate solution.

(b) Put 100 c.c. of your specimen into a beaker, boil it over a wire gauze, and keep it boiling gently for at least 10 minutes while you are carrying out (c).

(c) *Total hardness (i.e. temporary + permanent).* Using a pipette, transfer 25 c.c. of the unboiled specimen to the

¹ One for (say) every four students would be sufficient.

² A quantity for a class is conveniently prepared by dissolving 10 gm. of Castile soap (powder) in 660 c.c. of denatured alcohol, and making up to 1 litre with distilled water.

bottle. From a burette, run in the standard soap solution, 1 c.c. at a time, replacing the cork and shaking vigorously after each addition. Continue the addition of soap solution until a "permanent" lather is obtained, *i.e.* one which remains unbroken for a minute.

Suppose 14 c.c. were required. Repeat, running in 12 c.c. all at once, and then running in 0.5 c.c. at a time till the end point is reached. The shaking after each addition is essential.

(*d*) *Permanent hardness.* This is a continuation of (*b*). Pour the contents of the beaker into a graduate, rinse out with a little distilled water, and then add more distilled water until the volume is once more 100 c.c. Shake up well, transfer 25 c.c. of it to the bottle, and find the volume of soap solution required, exactly as in (*c*).

Q.1. The results may be set down and worked out as follows:—

Total hardness.

No. of c.c. required = $3 \sim 16.5^1$, = 13.5.

Permanent hardness.

No. of c.c. required = $4 \sim 10$ = 6.

Evidently the total hardness is measured by the number 13.5 and the permanent by the number 6.

∴ temporary hardness is measured by the number $13.5 - 6 = 7.5$.

∴ of the total hardness, the percentage which is permanent is

$$\frac{6}{13.5} \times 100 = 44,$$

and percentage which is temporary is $\frac{7.5}{13.5} \times 100 = 56$.

Additional Exercise

Take 50 c.c. of the specimen used in (*d*) (*i.e.* from which temporary hardness has been removed), and add 10 c.c. of the sodium carbonate solution provided. Divide into equal parts *A* and *B*, and bring *B* just to the boil, leaving *A* alone. Now find what volume of soap solution is required to form a permanent lather with *A* and *B* respectively.

Q.2. Results and conclusion?

¹ *i.e.* the actual burette readings were 3 and 16.5.

EXERCISE 32

SOME COMPOUNDS OF MAGNESIUM

Object.—To study some compounds of magnesium.

Apparatus and Materials.—(a) Bunsen; tripod stand; wire gauze; two beakers; evaporating dish; boiling tube; test-tube; dilute sulphuric acid; ammonium hydroxide. (b) Carbon dioxide generator (one to about every five students); litmus paper; magnesium carbonate “heavy”, 5 gm.; hydrated magnesium sulphate and chloride, say 5 gm. of each; soda crystals, 3 gm.; solution of ammonium chloride and of disodium phosphate.

Directions, etc.—1. *Preparation of Epsom salt.* Commercially this is prepared from magnesite, but as this is rather slow in dissolving we will use the “heavy carbonate”—really a basic carbonate having the formula $3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$.

Weigh out 5 gm. of this into a boiling tube, and add 25 c.c. of dilute sulphuric acid. If the carbonate has not completely dissolved in a minute or two, add just a little more acid.

Transfer to an evaporating dish and concentrate to about one-third the volume. Then pour back into the boiling tube and leave to crystallize out.

2. *Magnesium bicarbonate.* In the presence of water and carbon dioxide, calcium carbonate (insoluble) is turned into bicarbonate (soluble). We shall now show that magnesium carbonate undergoes a somewhat similar change.

Take 1 gm. of Epsom salt and 3 gm. of soda crystals ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$), and dissolve each in about 100 c.c. of water in a beaker. Add the second solution to the first. Q.1. What change do you notice? Q.2. Assuming that magnesium carbonate, MgCO_3 ¹, is formed, write the equation.

Now pass carbon dioxide through the liquid for a minute or two. Q.3. What happens? Q.4. Making the assumption

¹ Actually the change is somewhat more complicated; cf. Elem. Chem. Book II, p. 156.

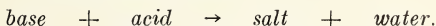
of Q.2, write the equation. Q.5. What would you expect to observe if you now heated the liquid?

Find by experiment if your supposition is correct.

3. *Action of heat on hydrated magnesium chloride, $MgCl_2 \cdot 6H_2O$.* Dissolve a little magnesium chloride in water, and try the effect on litmus. Q.6. Result?

Now heat a few grams of the substance in a test-tube. Notice the smell of the fumes given off when the substance has nearly reached the dry state, and also notice their effect on litmus. Q.7. What do they seem to be?

When all moisture has been driven off, let the tube cool a little, and then shake up with water. Q.8. How does the liquid affect litmus now? Q.9. It is commonly said with regard to neutralization that



Does the chemical change you have just studied bear any relation to this statement? Q.10. What term is used to describe such a change?

4. *Test for magnesium ion.* Dissolve a little magnesium sulphate in water, and add ammonium chloride, ammonium hydroxide and then disodium phosphate. Q.11. What do you notice? Q.12. Write the equation. (N.B.—The ammonium chloride is added only to prevent the precipitation of magnesium hydroxide, $Mg(OH)_2$, by the ammonia.)

EXERCISE 33

VALENCY OF MAGNESIUM

Object.—To find the valency of magnesium, assuming that its atomic weight is 24.1 [= 1].

Apparatus and Materials.—(a) *Beaker; sulphuric acid dilute.* (b) *Head of thistle funnel; burette; scale showing cm. and mm.; thermometer; magnesium ribbon (about 20 cm.).*

Preliminary Discussion.—If magnesium were univalent, then “Mg” would displace “H” from an acid, and 24.1 gm. magnesium would displace one gm. hydrogen.

If it were *divalent*, “Mg” would displace “2H”, and 24.1 gm. magnesium would displace *two* gm. hydrogen, and so on.

In fact, when we have found the number of grams of hydrogen displaced by 24.1 gm. of magnesium, we have found the valency; therefore, in the following experiment, we shall take a weighed quantity of magnesium, find the weight of hydrogen it displaces, and then calculate the weight of hydrogen that would be displaced by 24.1 gm.

Directions, etc.—First find the volume of the part BT of a burette (Fig. 18). This is easily done by running water into it from another burette.

Now arrange the apparatus shown in Fig. 18. At M is a quantity of magnesium ribbon of known weight, between .0300 and .0500 gm.¹ This is covered with the head of a thistle funnel, immersed in water in a beaker. The burette is filled with very dilute sulphuric acid², closed with the thumb and then placed over the thistle funnel. Hydrogen is quickly given off, the magnesium being completely dissolved in two or three minutes.

Q.1. Equation?

When the metal is wearing thin, it is apt to be carried to the top by the hydrogen and to stick to the side of the burette. If this happens, the burette should be shaken to prevent the magnesium being left “high and dry”. The readings to be taken are (i) volume of hydrogen, (ii) temperature of water in beaker, (iii) barometer, and (if the second method of calculation given below is adopted) (iv) height of column CL.

Q.2. Make a labelled sketch of your apparatus. **Q.3.** Record your readings and work

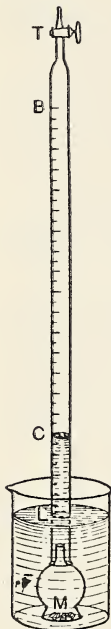


FIG. 18.

¹ *Note to teacher.* It is suggested that the class be told the weight of 100 cm. of the ribbon, and then each student can cut off a measured length.

² About 2N. Two parts water and one part ordinary “sulphuric acid dilute” will do very well.

out your result as in the following example, following either (i) or (ii) according to the instructions of your teacher.

Length of magnesium taken = 5.7 cm.

100 cm. weigh 0.68 gm. (given)

\therefore wt. of 5.7 cm. = $.0068 \times 5.7 = .0388$ gm.

Vol. of ungraduated part of burette = 4.2 c.c.

Burette reading at close of expt. = 34.7 c.c.

\therefore vol. of hydrogen = 38.9 c.c.

Temp. = 13°C. Barometer reading 75.2 cm.

(i) *Calculation, taking no account of pressure of water vapour, or of length of column CL.*

$$\begin{aligned} \text{Vol. of hydrogen at N.T.P.} &= 38.9 \times \frac{273}{286} \times \frac{75.2}{76} \\ &= 36.7 \text{ c.c.} \end{aligned}$$

\therefore (since 1 litre at N.T.P. weighs 0.09 gm.)

wt. of hydrogen = $.00009 \times 36.7 = .00330$ gm.

This is the weight displaced by .0388 gm. magnesium.

$$\begin{aligned} \therefore \text{wt. displaced by 24.1 gm. is} &= \frac{.00330}{.0388} \times 24.1 \\ &= 2.05. \end{aligned}$$

The valency is evidently *two*.

(ii) *Calculation, taking into account the pressure of water vapour, and length of column CL (= 15.6 cm.).*

If p be the pressure of the hydrogen in the burette, v the pressure of the water vapour, and c the pressure due to the column CL, we have

$$p + v + c = 75.2.$$

Now v is the maximum pressure of water vapour at 13°C. = 1.1 cm. (see Table, App.VII).

c is the pressure of (practically) a water column 15.6 cm. high = $15.6 \div 13.6$ or 1.2 cm. of mercury.

$\therefore p + 1.1 + 1.2 = 75.2$, whence $p = 72.9$ cm.

Thus, vol. of hydrogen at 72.9 cm. pressure and 13°C. is 38.9 c.c.

$$\therefore \text{vol. at N.T.P.} = 38.9 \times \frac{273}{286} \times \frac{72.9}{76} = 35.6 \text{ c.c.}$$

But at N.T.P. 1000 c.c. weighs 0.09 gm.

\therefore wt. of 35.6 c.c. = $.00009 \times 35.6 = .00320$ gm.

i.e. wt. of hydrogen displaced by .0388 gm. magnesium is .00320 gm.

$$\begin{aligned} \therefore \text{wt. displaced by 24.1 gm. is} &= \frac{.00320}{.0388} \times 24.1 \\ &= 1.99. \end{aligned}$$

This indicates a valency of *two*.

EXERCISE 34

ZINC AND SOME OF ITS COMPOUNDS

Object.—To learn more about zinc and some of its compounds.

Apparatus and Materials.—(a) *Bunsen; crucible tongs; pipeclay triangle; 4 test-tubes; tripod stand; retort stand; collar and clamp; dilute hydrochloric acid; ammonium hydroxide.* (b) *Crucible; combustion tube about 12" long and 5/8" int. diam.; Kipp's apparatus to supply H_2S (alternatively about 20 c.c. of strong H_2S solution); zinc sulphide and zinc sulphate, about 2 gm. of each; piece of granulated zinc, about 2 gm.*

For additional experiment.—*Solutions of sodium hydroxide and barium nitrate.*

Directions, etc.—We have already studied zinc to some extent. We know that it dissolves easily in hydrochloric acid, forming zinc chloride and hydrogen, while in Ex. 13 we found that it occupies a fairly high position in the electrochemical series. In this lesson we shall extend our knowledge.

1. *Zinc oxide.* Heat a bit of granulated zinc on charcoal in the tip of a blowpipe flame. *Q.1.* Can you make the zinc burn? *Q.2.* Can you see any "smoke", and if so, what is it likely to be? *Q.3.* What colour is zinc oxide (i) when hot, (ii) when cold?

2. *Action of acid on zinc sulphide.* Put about 1 gm. of zinc sulphide in a test-tube, add a few c.c. of dilute hydrochloric acid, and warm gently. *Q.4.* Write the equation for what you suppose has happened.

3. *Heating zinc sulphide in current of air.* By means of a paper trough (Fig. 12, p. 35) place about 1 gm. of zinc sulphide in an open combustion tube, two or three inches from one end. Arrange the tube in a sloping

position as shown in Fig. 19, and heat the sulphide strongly for about 5 minutes.

Q.5. Judging by the appearance, what should you say is coming from the upper end of the tube? Smell cautiously. *Q.6.* What can you smell?

Allow the substance in the tube to cool. *Q.7.* What colour is it (i) while still hot, (ii) after cooling?

Q.8. Can you suggest what the substance may be? Put half of the cool substance in a test-tube, add dilute

hydrochloric acid, and warm gently. *Q.9.* Can you smell anything now? *Q.10.* Write the equation showing the conversion of zinc sulphide into zinc oxide.

4. *Action of hydrogen sulphide.* Dissolve about 1 gm. of zinc sulphate in 10 c.c. of water, and divide into two parts.

(i) To one part add a few drops of hydrochloric acid, and then pass hydrogen sulphide through the solution (Kipp's apparatus).

(ii) To the second part add ammonia. A white precipitate of zinc hydroxide may be seen at first, but on adding a little more ammonia and shaking, this will disappear. Through the clear solution pass hydrogen sulphide.

Q.11. Do you get the same result this time, as when you passed hydrogen sulphide in 4. (i)? If not, account for the difference.

Notice the *colour* of zinc sulphide. Zinc is the only common metal which forms a white sulphide, and this fact is very useful when we are testing for zinc.

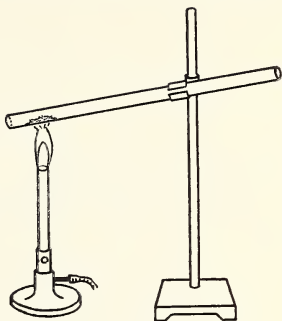


FIG. 19.

Additional Experiments

1. Take the other half of the substance obtained when zinc sulphide was heated in the combustion tube. Put it

in a test-tube, boil with water for two minutes, and then allow to settle. Pour off the clear liquid and test for a sulphate as described on p. 94.

Treat some of the original zinc sulphide in the same way (*i.e.* boil with water and test for a sulphate). Record your results and conclusion.

2. Make about 2 c.c. of a solution of zinc sulphate and add a little caustic soda solution. *Q.1.* What happens? *Q.2.* Write the equation.

Now add considerably more of the caustic soda solution and shake up. *Q.3.* What happens now?

EXERCISE 35

ALUMINIUM AND SOME OF ITS COMPOUNDS

Object.—To study aluminium and some of its compounds.

Apparatus and Materials.—(a) *Bunsen*; 3 test-tubes; boiling tube; 3 beakers; funnel (with stand), and filter paper; dilute hydrochloric acid and dilute sulphuric acid; ammonia; lime-water. (b) Charcoal block; blowpipe; two gas-jars with cover-slips; mortar and pestle; wooden splint; two pieces of finger bandage, each say 6" \times 2"; aluminium foil (2 gm.); powdered alum (20 gm.); powdered sodium carbonate (1 gm.); alizarin paste (2 gm.)¹; clay (10 gm.); litmus paper; 20% caustic soda solution (25 c.c.); 10% aluminium sulphate solution (1 c.c.); cobalt nitrate solution (a few drops).

Directions, etc.—The general physical properties of aluminium will be well-known, and so will the fact that after long exposure to air and moisture it is oxidized only very superficially.

1. *Action of hydrochloric acid.* Put some aluminium foil in a test-tube and add dilute hydrochloric acid. *Q.1.* What gas is evolved? *Q.2.* Assuming that aluminium is trivalent, write the equation.

2. *Action of sodium hydroxide.* Repeat, but this time using about 5 c.c. of caustic soda solution (instead of acid)

¹ If alizarin paste is not available a strong solution of litmus may be substituted.

and warming gently. Q.3. What gas is evolved now?

3. *Alum*. Dissolve a little alum in water. Dip a piece of litmus paper in it. Q.4. Result?

To a part of the solution add a little powdered sodium carbonate, and test for carbon dioxide as indicated in Fig. 17, p. 53. Q.5. Result? (The "acid" properties of alum are discussed in your text-book.)

4. *Aluminium hydroxide*. To another part of the solution of alum, add ammonia until it smells after shaking. The precipitate is aluminium hydroxide, $\text{Al}(\text{OH})_3$. Notice its curious gelatinous appearance. Filter, and after the liquid has drained through, pour boiling water on the precipitate to wash it.

Take out the filter paper gently, open it out, and scrape the hydroxide together with the edge of a wooden splint. Divide it into three parts, putting one part into each of two test-tubes, and the third part in a hole that has been scooped out in a charcoal block for blowpipe purposes.

To the first test-tube add a little dilute sulphuric acid, and to the second a little of the caustic soda solution supplied, in each case warming gently. Q.6. What happens?

N.B.—The result is due to the fact that in contact with a strong acid, aluminium oxide behaves like a basic oxide, while in contact with a strong base it behaves like an acidic oxide. Such an oxide is said to be *amphoteric*.

5. *Blowpipe test*. Heat the aluminium hydroxide strongly on the charcoal block. Then add two or three drops of cobalt nitrate solution and heat again. Q.7 What colour has the final product? This experiment provides a very fair confirmatory test for aluminium, but it is not quite characteristic, because certain phosphates and silicates yield a product of similar appearance.

6. *Dyeing*. In three beakers put respectively (i) a solution of alum, (ii) a solution of ammonia ("bench bottle" ammonia diluted with an equal volume of water), (iii) dilute caustic soda solution (the solution supplied,

diluted with twice its volume of water, into which a little alizarin paste has been stirred.

Now soak a piece of white cotton fabric (finger bandage will do) in the alum solution, and then dip it into the ammonia. This precipitates aluminium hydroxide within the fibres of the cloth. Put the latter into the litmus solution, and at the same time put in a piece of similar fabric which has not been treated with solutions (i) and (ii).

After a few minutes withdraw both pieces of fabric, and wash them under the tap. Q.8. In which case is the dye "fast", and is it fast even when boiled in water?

7. *Treatment of water.* Take two clean gas-jars and fill each about three parts full with water. Then rub up a little common clay with water in a mortar, and add an equal amount of the dirty water to each of the gas-jars. Shake both of them well.

To the first jar add about 2 c.c. of aluminium sulphate solution and say 10 c.c. of lime-water. Shake well once more. Put both jars on one side and observe them (i) a few minutes later, (ii) a few hours later. Q.9. Results?

EXERCISE 36

IRON AND SOME OF ITS COMPOUNDS

Object.—To observe the effect of heating and cooling steel; also to prepare ferrous sulphate and ferric oxide.

Apparatus and Materials.—(a) *Bunsen (Meker burner if possible); 3 beakers; filter funnel; retort stand and clamp; 2 test-tubes; glass rod; crucible tongs; dilute sulphuric acid.* (b) *Hard glass test-tube, fitted with cork and exit tube as shown in Fig. 20; filter paper; litmus paper; about 10 gm. iron filings (for this experiment, it is well to sieve these and reject the "dust"); piece of clock spring, about 3" long and 1/8" wide; powdered ferrous sulphate, about 10 gm.; magnesium ribbon, about 3"; barium nitrate solution.*

For Additional Experiment.—Flask fitted with thistle funnel, etc., as in Fig. 15, but no stand nor bunsen is required, and the down-tube is not so long; potassium permanganate, about 10 gm.; potassium ferrocyanide solution.

Directions, etc.—1. *Tempering of Steel.* Take the piece of clock-spring and heat it strongly in the bunsen (or Meker) burner. Then allow it to cool *slowly* (begin by holding it in the hot air just over the burner). Now try to bend it. Q.1. Does it bend easily, or is it springy?

Once more heat strongly, and cool it suddenly by plunging it into a beaker of water held only an inch or two to one side of the flame. Again try to bend it (near one end). Q.2. Result?

Heat it a third time, and cool it slowly as at first. Q.3. Does it bend easily now?

2. *Preparation of ferrous sulphate.* In a 250 c.c. beaker put 10 gm. of iron filings. Add 50 c.c. of dilute sulphuric acid, and if necessary warm a little on wire gauze so as to get the action to set in vigorously. Q.4. Write the equation.

Allow the action to proceed for at least 10 minutes (15 is better). Then let the liquid filter into a beaker or evaporating dish, and set the latter on one side until next lesson. By that time crystals will almost certainly have been deposited. (Do not attempt to evaporate the filtrate; you will probably only spoil it by oxidation.)

3. *Action of heat on ferrous sulphate.* While the solution of the iron filings is proceeding, prepare

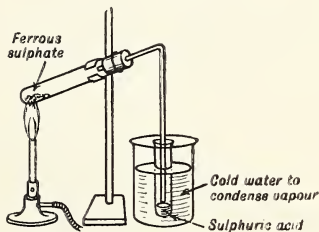


FIG. 20.

¹ If the time is short the test-tube may be heated without any attachments. In that case, however, it will not be possible to proceed with 3.

jeweller's rouge (very finely divided ferric oxide) as follows.

Fill the hard-glass test-tube almost a quarter full with powdered ferrous sulphate, and fix it up as indicated in Fig. 20. Heat as strongly as possible (use a Meker burner if one is available) for at least 5 minutes. Then allow to cool. *Q.5.* Does the ferrous sulphate appear to have been completely decomposed?

Rub a little of the residue between your thumb and finger. *Q.6.* Result? Examine the contents of the test-tube. *Q.7.* What can you smell?

Dilute the liquid in the test-tube with about three times its volume of water. Dip a glass rod in the diluted liquid and touch a piece of litmus with it. *Q.8.* Result?

Pour half of the liquid on a little magnesium ribbon contained in another test-tube, and test for hydrogen. *Q.9.* Results?

Test the remainder for a sulphate as described on p. 94. *Q.10.* Result? *Q.11.* If the liquid is both an acid and a sulphate, what is it?

*Additional Experiment**

Make a solution of potassium ferricyanide by passing chlorine through a solution of potassium ferrocyanide. (N.B. The chlorine is conveniently prepared by the action of dilute hydrochloric acid on potassium permanganate.)

EXERCISE 37

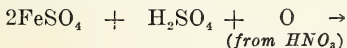
OTHER COMPOUNDS OF IRON

Object.—To learn more about ferrous and ferric compounds.

Apparatus and Materials.—(a) Bunsen; 4 test-tubes; hydrochloric acid; nitric acid; ammonium hydroxide. (b) Granulated zinc (1 gm.); ferric chloride (size of pea); solutions of potassium ferrocyanide and ferricyanide (the

latter freshly prepared); the beaker containing ferrous sulphate solution. etc., from last exercise.

Directions, etc.—1. Turning ferrous compounds into ferric. Into each of two test-tubes, pour about 2 c.c. of the mother liquor from the ferrous sulphate crystals made last lesson. To one of them add 5 drops of nitric acid and boil (notice the colour changes — very characteristic of the oxidation from ferrous salt to ferric). *Q.1.* What colour is (i) the untreated ferrous sulphate solution, and (ii) the solution that has been oxidized? *Q.2.* Complete the equation



To each test-tube add about 5 c.c. of ammonia solution. If after shaking it does not smell of ammonia, add more.

(*Caution.* The “nitric acid” test-tube often spurts violently when shaken with ammonia. See that it does not point towards anyone.)

Q.3. What is the colour of the precipitate in each case?
Q.4. Write the equations representing the action of ammonium hydroxide on ferrous sulphate and ferric sulphate respectively.

2. Ferric to ferrous. Dissolve a piece of ferric chloride the size of a pea in 5 c.c. of water. Add a fragment of zinc and about 1 c.c. of hydrochloric acid. Leave the mixture for two or three minutes. *Q.5.* Describe and account for any colour change that you notice. *Q.6.* Write the equation representing the action of nascent hydrogen on ferric chloride.

3. Distinctive tests. We have already seen that
 (i) in some cases at least, solutions of ferrous salts are green and solutions of ferric salts yellow or brown.
 (ii) ammonia gives a green precipitate with ferrous salts and a brown one with ferric salts.

There are some good distinctive tests. Pour off 2 or 3 c.c. of the ferrous sulphate mother liquor into each of two

test-tubes. Into each of two other test-tubes put 2 or 3 c.c. of ferric chloride solution. Then test as follows:

(iii) To the ferrous sulphate solution, and then to the ferric chloride solution, add a few drops of potassium ferrocyanide. *Q.7. Results?*

(iv) Repeat, but this time use potassium ferricyanide. *Q.8. Results?*

(v) We may also recall an experiment we did in Ex. 6 when we added ammonium thiocyanate to a ferric salt. This gave an intensely red coloration. When it is added to a ferrous salt (provided the latter is quite free from ferric) there is no change of colour. *Q.9. Complete the following table:*

	Colour of Solution	Effect of adding			
		Ammonia	Potassium ferrocyanide	Potassium ferricyanide	Ammonium thiocyanate
Ferrous salts					
Ferric salts					

EXERCISE 38

COPPER AND SOME OF ITS COMPOUNDS

Object.—To prepare two oxides of copper from a copper salt; and to work through a few of the chief “tests for copper”.

Apparatus and Materials.—(a) *Bunsen; beaker; tripod stand; wire gauze; evaporating dish; two test-tubes; ammonium hydroxide.* (b) *Kipp’s apparatus (generating H_2S), or alternatively a strong solution of H_2S ; platinum wire (or substitute); bright iron nail; powdered blue vitriol (a pinch); copper sulphate solution, about 7%, say 60 c.c.; about 25 c.c. of sodium hydroxide solution, 12% (for a class, add 300 c.c. of water to 500 c.c. of 5N sodium hydroxide); glucose, a few gm.*

Directions, etc.—In the course of other work we have incidentally experimented very considerably with copper

and its compounds. Thus, we are pretty certain to have observed the oxidation of the metal by air, and the reduction of black copper oxide by hydrogen (perhaps also by carbon monoxide and by carbon). When studying the oxides of nitrogen we learn incidentally how copper is acted upon by nitric acid, both dilute and concentrated — and this list of past studies in which copper plays a part could easily be lengthened.

In the present exercise we shall extend our knowledge of the compounds of copper.

1. *Black copper oxide* (CuO) *from a copper salt*. In a beaker put about 50 c.c. of the copper sulphate solution provided, and add about 20 c.c. of the sodium hydroxide solution. Q.1. What happens? Q.2. Equation?

Now boil, and continue to boil gently for a minute or two. Q.3. What change do you notice? Q.4. Assuming that the new substance formed is cupric oxide¹, CuO , write the equation.

Allow the precipitate to settle, and pour off the supernatant liquid. Add more water and repeat the process (*i.e.* boil for a minute, allow to settle, and pour off).

To obtain *pure* copper oxide we should continue to wash by decantation until the supernatant liquid no longer (i) turns litmus blue, or (ii) gives a precipitate with barium chloride. Q.5. Why these tests?

To save time, however, we will omit the complete washing. With the help of a glass rod, transfer the black substance to an evaporating dish, and heat on a wire gauze until steam ceases to be evolved. (During this operation the hydrated oxide loses its combined water, cupric oxide, CuO , being left).

2. *Red copper oxide* (Cu_2O) *from a copper salt*. If the preceding experiment is carried out in the presence of a suitable *reducing* agent, we may suppose that the black

¹ Actually it is a slightly hydrated oxide having the formula $(\text{CuO})_x \cdot \text{H}_2\text{O}$.

oxide loses its oxygen to the reducing agent ($2\text{CuO} \rightarrow \text{Cu}_2\text{O} + \text{O}$), cuprous oxide (Cu_2O) being formed.

To illustrate the reaction, dissolve 1 gm. of glucose (the reducing agent) in 5 c.c. of water. Then add about 2 c.c. of the sodium hydroxide solution, followed by 2 c.c. of the copper sulphate solution. Now heat. Red cuprous oxide at once makes its appearance, but we will not stop to obtain it in a pure condition.

3. Tests for the presence of copper.

(i) Notice that solutions of copper salts are usually blue.

(ii) Try the flame test as described on p. 87. Q.6. What colour do you observe?

(iii) Put some copper sulphate solution in an evaporating dish, and leave a bright iron nail in it for half a minute. Q.7. Result? Q.8. Write the equation.

(iv) Put a little copper sulphate solution in a test-tube and let a few bubbles of hydrogen sulphide pass into it. Q.9. Result? Q.10. Write the equation.

(v) To about 3 c.c. of copper sulphate solution add (a) a few drops of ammonia solution (b) a considerable quantity. Q.11. What happens?

EXERCISE 39

SILVER AND SOME OF ITS COMPOUNDS

Object.—To study silver and some of its compounds.

Apparatus and Materials.—(a) Bunsen; 2 test-tubes; beaker; tripod stand; wire gauze; silver nitrate solution (ordinary); sodium hydroxide solution. (b) Small sheet of glass, say 4" \times 4" (or a clean cover-slip would do); silver coin; wooden splint; mercury (one drop); glucose (about 1 gm.); powdered silver nitrate (1-2 gm.); strong solution of H_2S (a few drops); strong solution of silver nitrate (7 gm. to 100 c.c. of water—about 1 c.c.); dilute ammonia solution (one-fifth usual strength).

For additional experiment.—Balance, etc.; dry test-tube; about 1 gm. silver nitrate (powdered).

Directions, etc. —1. *Tarnishing of silver.* Pour a few drops of a strong solution of hydrogen sulphide on a silver coin, and after a few seconds rinse under the tap. *Q.1.* What do you suppose the dark stuff is? *Q.2.* Can you account for the tarnishing of silver ornaments, etc.?

2. *Electromotive series.* In Ex. 13 we saw that both silver and mercury are lower than copper in the electromotive series. The following experiment shows that silver is lower than mercury.

Pour about 1 c.c. of the given strong solution of silver nitrate on a clean piece of glass. Put a very small drop of mercury on the glass, and push it just inside the edge of the little pool of silver nitrate. The glass should be in a position where it can remain quite undisturbed till the end of the lesson. Examine the little pool from time to time. The feathery crystals that form are metallic silver.

3. *Silver oxide easily reduced.* As silver is lower even than mercury in the electromotive series, we should expect that its oxide would be very easily reduced.

(i) To about 5 c.c. of silver nitrate solution in a test-tube add a little sodium hydroxide solution. This gives a brown precipitate of silver oxide ($2\text{AgNO}_3 + 2\text{NaOH} \rightarrow 2\text{NaNO}_3 + \text{Ag}_2\text{O} + \text{H}_2\text{O}$). Now add the specially diluted ammonia solution provided, shaking from time to time, until the precipitate is *just* dissolved.

To reduce this solution of silver oxide to silver, add a little glucose solution, shake up and warm gently (it is best to hold the test-tube in a beaker of hot water). A silver mirror should be obtained.

ii) Heat a little powdered silver nitrate in a clean test-tube, and hold a glowing splint just inside the tube. Brown fumes are given off, and in the first instance silver oxide is left ($4\text{AgNO}_3 \rightarrow 2\text{Ag}_2\text{O} + 4\text{NO}_2 + \text{O}_2$). This oxide is itself decomposed however, so that the final product is silver. *Q.3.* How do you know that nitrogen peroxide was evolved? *Q.4.* What happened to the splint, and why?

Additional Experiment

Percentage of silver in silver nitrate. Weigh a clean dry test-tube as accurately as possible, and then add about 1 gm. of silver nitrate and weigh again. Use a paper trough (Fig. 12, p. 35) to secure that the substance shall all be deposited at the *bottom* of the test-tube.

Make a paper test-tube holder and begin to heat the nitrate, gently at first (until it has completely melted) and then as strongly as possible. Continue to heat for three or four minutes after all brown fumes have ceased to be evolved.

Sometimes a little of the molten nitrate "creeps" up the sides of the test-tube, and in that case it will be necessary to give the upper part of the tube its turn of being heated.

The nitrate is completely decomposed, only silver being left in the tube. When the latter has cooled down, weigh again. *Q.1.* Enter all weighings and work out the percentage of silver. *Q.2.* Calculate the theoretical result and compare.

EXERCISE 40

HALOGEN COMPOUNDS OF SILVER

Object.—To study the halogen compounds of silver.

Apparatus and Materials.—(a) 4 test-tubes; ammonium hydroxide; dilute nitric acid. (b) Potassium chloride, bromide, and iodide, about 1 gm. of each; silver nitrate solution; solution of "hypo" of about 10% strength, say 20 c.c.

Directions, etc.—1. *Silver chloride.* Dissolve a few grains of potassium chloride in about 3 c.c. of water in a test-tube, and add about 3 c.c. of silver nitrate. *Q.1.* What happens? *Q.2.* Complete the equation $\text{KCl} + \text{AgNO}_3 \rightarrow$.

Shake the precipitate so as to form a fairly uniform suspension, and then pour about half of it into another test-tube. To one portion add ammonia solution — enough to half fill the test-tube — and shake. *Q.3.* What happens?

To the other portion add dilute nitric acid — also enough to half fill the test-tube — and shake. *Q.4.* What happens?

2. *Silver bromide.* Proceed as already described, but using potassium bromide instead of potassium chloride.

3. *Silver iodide.* Proceed as before, but using potassium iodide.

(N.B.—Silver chloride, bromide, and iodide are all insoluble in dilute nitric acid. This part of the test serves to distinguish them, not from one another, but from certain other precipitates, *e.g.* silver phosphate, which are *soluble* in nitric acid.)

Q.5. Could you distinguish between silver chloride, bromide, and iodide by their colour? *Q.6.* Could you distinguish them by their solubility in ammonia?

4. *Effect of "hypo" on silver bromide.* Prepare a fresh quantity of silver bromide (by adding silver nitrate to potassium bromide). Shake up, boil, allow the precipitate to settle, and pour off the clear liquid.

Now half fill the test-tube with the given solution of "hypo" and shake up. *Q.7.* What happens? *Q.8.* Given that the soluble compound produced has the formula NaAgS_2O_3 , attempt the equation.

(N.B.—The process of "fixing" in photography depends on the solubility of silver bromide in a solution of "hypo".)

EXERCISE 41

SILVER FROM A COIN

Object.—To prepare a specimen of pure silver from a 10-cent piece.

Apparatus and Materials.—(a) Bunsen; boiling tube; test-tube; glass rod; tripod stand; wire gauze; small evaporating dish; nitric acid; hydrochloric acid dilute; ammonium hydroxide. (b) Charcoal block; blowpipe; 10-cent piece (one to 5 students).

Directions, etc.—Put the coin in a boiling tube, add 10 c.c. of water and then 10 c.c. of nitric acid. Warm a

little until a vigorous action sets in, and when the coin has completely disappeared the solution may be divided among the five students concerned. From this point the directions apply to individual workers.

Q.1. What colour is the solution? *Q.2.* Can you suggest the reason?

Add a few c.c. of dilute hydrochloric acid. *Q.3.* What happens? *Q.4.* Write the equation.

Boil (this helps the precipitate to settle). Pour off a little of the supernatant liquid into a test-tube and add *excess* of ammonia (*i.e.* the liquid must smell of ammonia after shaking). *Q.5.* What change do you notice? *Q.6.* What does this indicate? (If necessary, refer back to p. 70).

Pour off the remaining supernatant liquid. Then quarter fill the tube with water, boil, allow the precipitate to settle and pour off the clear liquid. Repeat this "washing by decantation" twice more.

Now transfer the silver chloride to a small evaporating dish and heat gently over a wire gauze till the chloride is dry. Add about an equal bulk of anhydrous sodium carbonate, mix well with a glass rod, and transfer to a hole scooped out in a charcoal block. Now heat strongly with a blowpipe. It is much harder to get a bead of silver than of lead, because of the much higher melting point. However in three or four minutes you should get a sort of "splash" of silver, and if your blowing is good you can make this run together into a bead.

EXERCISE 42

OXIDES OF LEAD

Object.—To study the three common oxides of lead.

Apparatus and Materials.—(a) *Bunsen; three test-tubes¹; boiling tube; evaporating dish; filter funnel; retort*

¹ Test-tubes with damaged tops may well be used for these experiments. A test-tube is almost invariably spoilt by the heating in it of an oxide of lead, or of lead nitrate.

stand with ring; wire gauze; hydrochloric acid; nitric acid dilute. (b) Filter paper; wooden splint; litmus paper, lead monoxide and peroxide, a few gm. of each; red lead, about 30 gm.

Preliminary Note.—The three common oxides of lead are lead monoxide or litharge, PbO ; red lead, Pb_3O_4 and lead peroxide, PbO_2 . Beginning with red lead, we will try to obtain litharge by loss of oxygen, and lead peroxide by gain of oxygen.

Directions, etc.—1. *Action of heat on red lead.* Put red lead into a test-tube to a depth of about an inch and heat. Q.1. Is any oxygen evolved (glowing splint test)? Q.2. What colour is the residue (i) when hot, (ii) when cold? Q.3. Write the equation for the chemical change that has taken place.

2. *Action of nitric acid on red lead.* (i) Arrange a glass funnel, etc., all ready for filtering, with a boiling tube in position for receiving a filtrate.

(ii) Put 50 c.c. of dilute nitric acid into an evaporating dish and put this on a wire gauze over a bunsen. Add 20 gm. of red lead rather slowly to the acid. Bring the mixture to the boil, stirring all the time, and keep boiling for a minute.

The brown powder produced (PbO_2) settles fairly easily. Without giving the clear liquid time to cool much below boiling point, pour it through the filter.

The brown powder must be washed at least twice by decantation; so half fill the dish with water, boil, allow the powder to settle, and pour off the clear liquid. Then repeat this washing process. Afterwards heat the dish *gently* until the powder is dry.

Now examine the boiling tube. Q.1. What do you notice? Q.2. Assuming that the brown powder is lead peroxide, PbO_2 , complete the equation $\text{Pb}_3\text{O}_4 + 4\text{HNO}_3 \rightarrow$.

Heat some of the brown powder in a test-tube¹. Q.3. Is oxygen evolved? Q.4. What appears to be left in the test-

¹ Brown fumes are usually evolved, owing to the presence of some lead nitrate. The fact is that washing twice by decantation (as directed above) is not really sufficient, but time is a consideration.

tube? *Q.5.* Write the equation representing the action of heat on lead peroxide.

3. Action of hydrochloric acid on the three oxides. Heat a little red lead in a test-tube with hydrochloric acid, and smell *cautiously*; also hold a piece of damp litmus paper to the mouth of the tube. *Q.6.* What gas is coming off? *Q.7.* Equation?

In a similar way, examine the action of hydrochloric acid on lead monoxide and lead peroxide. *Q.8.* Record your results. *Q.9.* Write the appropriate equations.

EXERCISE 43

OTHER COMPOUNDS OF LEAD

Object.—To study some other compounds of lead.

Apparatus and Materials.—(a) *Bunsen*; four test-tubes; boiling tube; hydrochloric acid; sulphuric acid dilute. (b) *Kipp's apparatus* for H_2S (or a strong solution of the gas); wooden splint; lead nitrate and red lead (a few gm. of each); potassium chromate solution.

Directions, etc.—1. *Preparation of lead chloride.* Heat 3 or 4 gm. of red lead in a boiling tube with hydrochloric acid until chlorine ceases to be evolved. Let the buff-coloured product settle, and pour off the supernatant liquid.

Now one-third fill the tube with water and boil. Allow only two or three seconds for the undissolved portion to settle, and then pour the clear liquid into a test-tube. Cool under the tap. *Q.1.* What happens?

2. *Action of heat on lead nitrate.* Heat a few grams of lead nitrate in a test-tube. Hold a glowing splint just inside the tube. *Q.2.* What two gases are being evolved?

Allow the tube to cool. *Q.3.* What solid substance appears to be left? *Q.4.* Write the equation showing the action of heat on lead nitrate.

3. *Production of lead sulphate.* Dissolve a little lead nitrate in half a test-tube of water, and use a third of the

solution for this experiment, and a third for each of the next two.

To the first of the three portions add *one drop* of dilute sulphuric acid. Q.5. What happens? Q.6. What can you say about the solubility of lead sulphate? Q.7. Write the equation showing the action of sulphuric acid on lead nitrate.

4. *Production of lead chromate.* To the second portion of the lead nitrate solution add a little potassium chromate solution. Q.8. What happens? Q.9. Equation?

5. *Production of lead sulphide.* Through the third portion pass hydrogen sulphide (or add a strong solution of the gas). Q.10. Equation?

EXERCISE 44*

ACETYLENE

Object.—To prepare acetylene and examine its properties.

Apparatus and Materials.—(a) Beaker; funnel; tripod stand. (b) 3 gas-jars (2" internal diam.), and cover slips; pneumatic trough (or sink may be used); glass tube about 8" long; piece of lead foil about 4" \times 3"; filter paper; small piece of gummed paper; calcium carbide, 6 gm.; acetone, about 20 c.c.

Directions, etc.—1. *Preparation of gas.* Weigh out 6 gm. of calcium carbide and put it in the middle of the lead foil. Then fold this over as indicated by the dotted lines so as to make a packet about 1½" \times 1", the exposed ends being on the upper side. Through the upper surface make about a dozen jabs with your pocket knife.

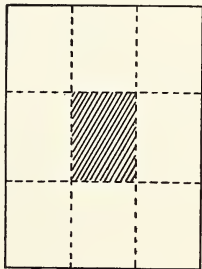


FIG. 21.

Run water into the trough to a depth of $1\frac{1}{2}$ " or so; also fill the gas-jars and invert them in the trough. Now place the packet of carbide in the trough, "holes" side uppermost; and collect two jars of gas, and half of a third jar. Leave the last half-jar in the trough, and with a piece of gummed paper mark the level of the water in it. Then open out the packet, transfer it to a beaker containing about 50 c.c. of water, and place this in the fume cupboard. Q.1. Write the equation for the action that has been taking place.

2. *Some properties of the gas.*

Q.2. Record the appearance and smell of the gas (the smell, however, is really due to the presence of phosphine, PH_3).

Still leaving in the trough the jar that is only half filled, shake it vigorously so that splashes of water come freely into contact with the gas. Q.3. Is acetylene soluble in water?

To a second jar of the gas add about 20 c.c. of acetone. Replace the cover-slip; shake up, and then open the jar under water. Q.4. What happens? What would you say about the solubility of the gas in acetone?

To the third jar of the gas apply a light. Q.5. What happens?

3. *Examination of liquid.* Q.6. What substance would you expect to be present in water that has reacted with calcium carbide?

Make a rough test of the correctness of your answer as follows.

Stir up the contents of the beaker which you left in the fume cupboard, and filter about 20 c.c. into another beaker. Dip a piece of litmus paper into the filtrate. Q.7. What happens?

Breathe into the filtrate through a glass tube. Q.8. What happens?

EXERCISE 45*

STARCH

Object.—To study some properties of starch.

Apparatus and Materials.—(a) *Bunsen* (two, if available); 3 beakers; 6 test-tubes; tripod stand; wire gauze; evaporating dish; deflagrating spoon; glass rod; lime-water; dilute sulphuric acid. (b) Gas-jar; cover-slip; thermometer; powdered starch (say 5 gm.); about 20 c.c. of copper sulphate solution (about 7%), and the same of sodium hydroxide solution (about 12%; for a class, add 150 c.c. of water to 250 c.c. of 5N sodium hydroxide); malt extract (e.g. Liebig's), 2 or 3 gm.; dehydrated copper sulphate, about 1 gm.

Review of previous knowledge.—From seeing “starch” (i.e. the liquid) made at home, we already know something about it. We know that it is insoluble in cold water, but forms a curious sort of solution when added to boiling water. Actually the starch consists of very tiny granules. In contact with boiling water the cell walls burst, and the contents pass into colloidal solution (*Elem. Chem. II*, p. 97). We shall presently have to make some of this starch solution for use in our experiments.

Again, from Ex. 16, we know that when iodine is added to starch, a blue colour is produced. This gives us an extremely sensitive test for starch.

Directions.—1. *Preparing starch solution.* About three-parts fill a beaker with water, and boil it over wire gauze. Meanwhile, put a pinch of starch powder into an evaporating dish, and add just enough water to make it into a “cream” when stirred up with a glass rod. Pour this liquid into the boiling water (at this stage it will easily boil over if you are not careful), and keep it boiling gently for another minute. Then put it on one side for the experiments that follow.

2. *Beginning of experiments with malt, etc.* (i) Heat half a beaker of water to about 65°C. (not more). Now

about one-third fill a test-tube with starch solution, add a little malt extract, and surround the test-tube with the hot water in the beaker.

(ii) Take a second test-tube and repeat the preceding work, but adding a little saliva, instead of malt extract, to the starch solution. Put this test-tube beside the other, surrounded with hot water.

(iii) Put about 100 c.c. of starch solution in a beaker, and add say 10 c.c. of dilute sulphuric acid. Boil over a wire gauze, and leave it boiling very gently.

The starch + malt, starch + saliva, and starch + sulphuric acid, will all be wanted presently.

3. *Effect of heat.* Heat a few grams of starch in a test-tube, gently at first, then strongly. If any liquid is produced, let a drop of it fall on a "crumb" of dehydrated blue vitriol. Q.1. Record your observations. Q.2. Can you produce evidence that starch contains (i) carbon, (ii) hydrogen, (iii) possibly, oxygen? Q.3. Why is one less certain about the oxygen?

Put some starch in a deflagrating spoon. Heat it with the bunsen till it takes fire, and then lower it into a gas-jar. Afterwards shake up with lime-water. Q.4. Results and conclusion?

4. *Will starch reduce copper sulphate?* In Ex. 38 we saw that in the presence of caustic soda, glucose will reduce copper sulphate to cuprous oxide.

Take a little starch solution in a test-tube, add about an equal volume of caustic soda solution, and then a little of the copper sulphate solution. Now heat, and observe whether any red cuprous oxide is formed. Q.5. Does starch behave like glucose in reducing copper sulphate?

5. *Effect of malt, etc.* Now apply the same treatment (i.e. adding caustic soda and copper sulphate, followed by heating) to the starch which has been treated with (i) malt,

(ii) saliva, (iii) sulphuric acid. Q.6. Results? Q.7. Can you see one reason why it is important to chew food properly before swallowing it?

EXERCISE 46

SUGARS

Object.—To illustrate a process used in the purification of cane sugar; also to study some properties of cane sugar and of dextrose (glucose).

Apparatus and Materials.—(a) Bunsen; beaker; stirring rod; tripod stand; wire gauze; funnel; boiling tube; three test-tubes; lime-water; sulphuric acid dilute; silver nitrate solution. (b) Filter paper; cane sugar (about 15 gm.); black molasses and animal charcoal (about 5 gm. of each); honey (1 gm.); litmus solution (a few drops); ammonia solution (bench bottle solution diluted to five times its volume), say 30 c.c.

Directions, etc.—1. *Purification of cane sugar.* Put about 50 c.c. of water in a beaker, and dissolve in it 2 or 3 c.c. of black molasses. Note the colour of the solution. Now add about 5 gm. of animal charcoal, and boil for a minute or two. Filter the solution. Q.1. In what way has it changed?

2. *Effect of sulphuric acid.* Put about 10 gm. of sugar in a beaker and add 7 c.c. of water. Heat to dissolve the sugar, and then pour 10 c.c. of sulphuric acid into the warm solution. Q.2. What happens, and how do you account for the result?

3. *Reducing action of sugars.* Prepare an ammoniacal solution of silver oxide as described on p. 71, but a larger quantity will be required, so start with about 15 c.c. of silver nitrate solution.

(i) The reduction of silver oxide to silver, by means of glucose, has already been shown (p. 71).

(ii) Repeat with a solution of honey.

(iii) Repeat with a solution of cane sugar.

(iv) Prepare about 10 c.c. of another solution of cane sugar and add to it about 1 c.c. of dilute sulphuric acid. Heat gently for about a minute (not quite to boiling) and put it on one side for a few minutes.

We must now get rid of the excess of acid. To do this put in a few drops of litmus solution, and add the special ammonia solution, with constant shaking, until the litmus is *just* turned blue.

Now add the silver oxide solution. *Q.3.* Summarize the results of Expts. 3 (i) - (iv), especially noting whether the treatment with sulphuric acid made any difference to the cane sugar.

EXERCISE 47*

ALCOHOL

Object.—To prepare ethyl alcohol from glucose, and to examine some of its properties.

Apparatus and Materials.—(a) *Bunsen; two beakers; retort stand with two rings; collar and clamp; wire gauze; deflagrating spoon; three test-tubes; taper; lime-water; sulphuric acid.* (b) *Flask (500 c.c.), fitted with cork and tubes as shown in Fig. 22; mortar and pestle; thermometer; gas-jar and cover-slip; glucose, say 30 gm.; yeast, 7 gm.; alcohol, fairly pure, about 10 c.c.; potassium dichromate, a pinch; common salt, camphor and iodine — a few grains of each.*

Preliminary Note.—In the interests of logical order, the complete “Preparation” has been put before “Properties”. In practice, it will be necessary to set up the apparatus of Fig. 22 in a first lesson, carrying out as many of the other experiments as there is time for. During a second lesson, the experiment indicated by the dotted lines of Fig. 22 would be carried out, and the experiments on “Properties” completed.

Directions, etc.—1. *Preparation.* Half fill a 500 c.c. flask with water, and in this dissolve 25 gm. of glucose. Then put about 7 gm. of yeast in a mortar and reduce it to a liquid condition by adding a little glucose and

rubbing for a minute with the pestle. Add this yeast to the glucose solution.

Fit up the flask as shown in Fig. 22, the delivery tube passing into lime-water in a beaker. The tube AB should be about 18 inches long. The flask must be kept at 25° to 30°C . for at least 24 hours¹. By the side of the beaker place a second "control" beaker containing lime-water, so that you can see if there is any change produced other than that due to atmospheric carbon dioxide. Q.1. Make a labelled sketch of your apparatus.

Examine your apparatus at the end of about (i) an hour, (ii) 24 hours. Q.2. What do you notice?

After 24 hours (or longer) remove the bent tube ACA', and by means of a clamp support AB in the position A'B'. Now heat the flask, and from time to time apply a lighted taper to B'. Q.3. What happens? Q.4. Write an equation expressing the change undergone by the glucose.

2. *Some simple physical properties.* Examine a little of the alcohol provided. Q.5. Colour and smell?

Mix a few drops of it with a few drops of water. Q.6. Does it dissolve?

3. *Effect of burning.* Put a little on a deflagrating spoon, apply a light, and allow the liquid to burn in a

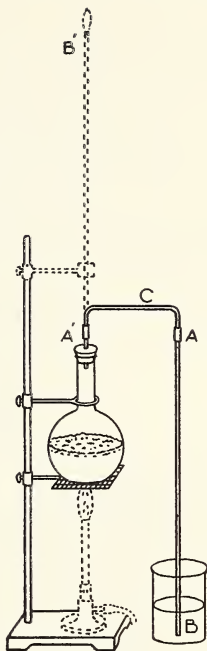


FIG. 22.

¹ A fume cupboard containing a bunsen burning with a small flame will serve; or the flask may be placed near heating pipes, etc.

gas-jar. Then shake up with lime-water. *Q.7.* What happens? *Q.8.* What element must be present in alcohol?

4. *Solvent action.* Put two or three grains of common salt in one test-tube, of camphor in another, and of iodine in a third. Add about 1 c.c. of alcohol to each, and leave till the end of the lesson, giving a shake now and then. *Q.9.* Do these substances dissolve in alcohol?

5. *Production of Aldehyde.* To about 1 c.c. of alcohol in a test-tube add a pinch of potassium dichromate and about 1 c.c. of sulphuric acid. Then heat gently. *Q.10.* Do you see anything which tells you that the dichromate is being reduced?

The curious smell noticed is that of *aldehyde*, C_2H_4O . *Q.11.* Equation? (N.B.—You may simplify matters by writing “O” instead of “ $K_2Cr_2O_7 + H_2SO_4$ ”.)

EXERCISE 48*

ACETIC ACID

Object.—To prepare acetic acid, and examine some of its properties.

Apparatus and Materials.—(a) *Bunsen; retort stand; collar and clamp; tripod stand; wire gauze; beaker; 3 test-tubes; glass rod; sulphuric acid.* (b) *Retort; thistle funnel; litmus paper; fused sodium acetate, coarsely powdered (about 20 gm.); sodium bicarbonate (a pinch); alcohol (about 1 c.c.).*

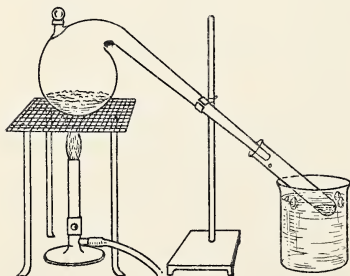


Fig. 23.

Directions, etc. — Arrange your apparatus as shown in Fig 23. Then put about 20 gm. of fused sodium ace-

tate into the retort and cover it with sulphuric acid, adding the acid by means of a thistle funnel. Heat the mixture, and collect the acetic acid which distils over. *Q.1.* Make a labelled sketch of your apparatus. *Q.2.* Write the equation for the reaction. (*Hint.*—Sodium acetate is $\text{Na} \cdot \text{C}_2\text{H}_3\text{O}_2$.) *Q.3.* Colour and smell of the acid?

Dilute a little of the acid with about 15 times its volume of water, and taste a drop of the diluted liquid (use glass rod). *Q.4.* Result? *Q.5.* Effect of diluted liquid on litmus?

Add a stronger solution of the acid (acid-1 part, water-3 parts) to a little sodium bicarbonate in a test-tube. *Q.6.* Result? Equation?

Add a little acetic acid to some alcohol in a test-tube, and then add sulphuric acid,—about 1 c.c. of each liquid. Heat gently. *Q.7.* Smell? Equation?

EXERCISE 49*

SOAP

Object.—To prepare soap, and to show how a fatty acid may be obtained from it.

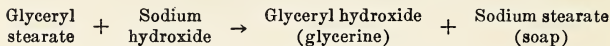
Apparatus and Materials.—(a) *Bunsen; two beakers; boiling tube; test-tube; hydrochloric acid.* (b) *Balance, etc.; graduate; mortar and pestle; common salt (10 gm.); lard or tallow (4 gm.); sodium hydroxide (2 gm.); wood spirit (20 c.c.)*

Directions, etc.—Set a beaker of water on wire gauze, to boil. This will be wanted presently.

Put 2 gm. of sodium hydroxide in a mortar and grind it to rather small bits. Add 20 drops of water and continue grinding, making a sort of cream. Then add 20 c.c. of wood spirit and stir. The object of all this is to obtain a solution of sodium hydroxide in spirit.

In a boiling tube put 4 gm. of lard or tallow, and melt it by heating it gently in the bunsen flame. Then add the solution of sodium hydroxide. Put the test-tube in the

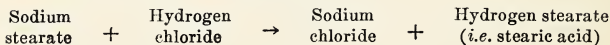
hot water in the beaker, and keep shaking it. The layer of fat can be seen on the surface, but after 3 or 4 minutes this fatty layer disappears, hydrolysis having taken place.



If water had been used (as in ordinary soap manufacture) hydrolysis would only have taken place very slowly, because the water would not have served as a solvent for the fat.

Continue the heating for an extra minute, and then quickly¹ empty the contents of the boiling tube into another beaker. On cooling, it hardens. Add 100 c.c. of water and heat. It soon dissolves.

Now pour about a quarter of the liquid back into the boiling tube, and add a little hydrochloric acid. This at once liberates the fatty acid—



The liquid has a white appearance, owing to the fatty acid being dispersed in minute drops (*cf.* milk). On boiling, the droplets run together, and an oily layer forms on the surface. It hardens on cooling.

Now prepare a strong solution of salt by adding 10 gm. of salt to 30 c.c. of water and stirring. Add this salt solution to the liquid in the beaker. The soap is thus "salted out" and appears in the form of curds. Pick out a little of this curd with your fingers, put it into a test-tube, add water, and heat. It should lather freely.

Q.1. Write a short account of your experiments, and of the results obtained.

¹ If the liquid is allowed to solidify, it is not very easy to re-melt if tallow was used in the first instance. If lard is used there is no trouble.

EXERCISE 50

FLAME TESTS

Object.—To examine the flame colorations given by salts of sodium, potassium, lithium, calcium, strontium, barium, and copper, respectively.

Apparatus and Materials.—(a) *Bunsen; evaporating dish; dilute hydrochloric acid.* (b) *Platinum wire in glass holder¹; watch glass; blue glass; a few grains of each of the following salts:—sodium nitrate, potassium nitrate, lithium chloride, calcium carbonate, strontium chloride, barium chloride, and copper sulphate.*

Directions, etc.—Bend the platinum round so as to form a small loop near the end. Put some dilute hydrochloric acid into a clean evaporating dish, dip the wire into this and then hold it in the bunsen flame. Do this several times until the wire is clean, and no longer gives any colour to the flame.



FIG. 24.

Now put a few grains of sodium nitrate into a watch glass, moisten it with dilute hydrochloric acid, and dip the wire into it. Hold the wire in the flame. *Q.1.* What colour do you notice?

The salt is moistened with hydrochloric acid in order that a little chloride may be formed. Chlorides are comparatively volatile, and so are specially suitable for flame tests.

Actually all sodium salts give a good flame reaction even without the acid, so in this case it is not really necessary to add it. In the case of salts of many other metals, however, the presence of hydrochloric acid makes a great difference, and so it is well to form the habit of adding it every time.

Repeat with potassium nitrate. *Q.2.* Colour?

Repeat, this time observing the flame through one or two thicknesses of blue glass. *Q.3.* Colour?

Potassium salts often contain small traces of sodium salts as an impurity, and the characteristic potassium flame is then masked

¹ A common and admirable method of keeping a platinum wire clean when not in use is indicated in Fig. 24.

by the strong yellow due to the sodium. By using blue glass we are able to cut off these yellow rays.

Carry out further flame tests with the remaining salts. Q.4. Record your results in tabular form, including those previously obtained. Q.5. Do you find any difficulty in distinguishing between calcium salts and strontium salts by the flame tests?

Repeat the work so far as salts of these two metals are concerned, but this time observe the flame through blue glass. Q.6. Can you now suggest a better means of distinguishing between calcium salts and strontium salts?

EXERCISE 51¹

QUALITATIVE ANALYSIS — CATIONS

Introduction to Simple Qualitative Analysis.—An important part of the work of the analyst consists in the identification of *salts*. Now a salt is the substance formed when the hydrogen of an acid is replaced by a metal, and as there are hundreds of acids and perhaps fifty or sixty metals, it is evident that the number of different salts is very large indeed.

However, in what follows we shall deal only with the salts derived from seven acids and thirteen metals (counting “ammonium” as one). This will greatly simplify the work, but at the same time it will give us some idea of the general principles of analysis.

A salt when dissolved in water gives rise to two ions, as we have already seen in Elementary Chemistry, Book II, Chapter IV, Copper sulphate, for instance, gives us copper ion Cu^{++} , and sulphate ion SO_4^{--} , known respectively as the *positive ion* or *cation* (because in electrolysis it appears at the cathode) and the *negative ion* or *anion*. They are often referred to simply as the “metal” (or “base”), and the “acid” (or “acid radical”), respectively. It does not matter very much which we identify first, but we will begin with the cation. In the little scheme outlined below, it is understood that this is one of the following fifteen:—silver, lead, mercury (-ous), mercury (-ic), copper, antimony, aluminium, iron (ferrous), iron (ferric), zinc, calcium, magnesium, sodium, potassium, ammonium. It is understood also that only a single salt is being identified.

Practical directions will be given in ordinary type, and notes, explanations, etc., in smaller type.

¹ Actually the material of this exercise is sufficient for several lessons, but a division would be somewhat artificial.

In the course of the “**Directions, etc.**” the usual incidental questions *Q.1, Q.2*, etc., will not be set, but a record of the practical work should certainly be kept. A three-column arrangement has been found to work satisfactorily, the notes appearing something like this:

<i>Experiment</i>	<i>Result</i>	<i>Inference</i>
1. Dissolved in water and tested with litmus.	Litmus turned red.	Hydrogen ion present (perhaps an acid, or a salt consisting of weak base combined with strong acid).
2. Added dil. HCl.	No pp.	Ag, Pb, and Hg' absent.
3. Passed H ₂ S through acid solution.	Black pp.	Cu or Hg'' present.
4. Boiled the washed pp. with dilute HNO ₃ . etc.	Pp. dissolved, a blue solution being formed. etc.	Copper present. etc.

We are now in a position to proceed with the practical work.

Object.—To learn how to identify the “metal” present in a given soluble salt.

Apparatus and Materials.—(a) *Bunsen; tripod stand; wire gauze; evaporating dish; rack of test-tubes; funnel.* (b) *Filter papers; blue glass; wood splint; platinum wire or substitute; litmus paper; usual reagents for qualitative analysis (see Appendix III); suitable salts (or solutions of salts) for identification.*

Directions, etc.—1. Dissolve in water (if the salt is not already dissolved) and test with litmus paper. If the latter goes red, *hydrogen ion* is present.

We must not conclude, however, that the substance in solution is necessarily an acid. It may be a salt, in which a weak base is combined with a strong acid (*e.g.* copper sulphate or ferric chloride).

2. Add *two or three drops* of dilute hydrochloric acid. If this gives a precipitate, add more acid until all the metal is precipitated.

A white precipitate indicates *silver, lead or mercury (-ous)*. To find out which:—

(i) Divide the precipitate into two parts. Add ammonium hydroxide to one part and dilute nitric acid to the other.

If it dissolves in the first case and not in the second, *silver* is present.

(ii) If the precipitate turns grey-black on the addition of ammonia, but does not dissolve, *mercury* (-ous) is present.

(iii) To the original solution add a solution of potassium chromate. A yellow precipitate of lead chromate indicates *lead*.

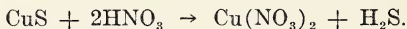
3. If silver, lead and mercury (-ous) are absent, pass hydrogen sulphide through the solution containing hydrochloric acid (or add a solution of hydrogen sulphide).

A black precipitate indicates *mercury* (-ic) or *copper*; an orange one *antimony*.

Filter, and pour boiling water on the precipitate to wash it. With a wood splint, transfer a little to a test-tube.

(i) If it is black, try to dissolve it in a little boiling dilute nitric acid. If it is insoluble, *mercury* (-ic) is present.

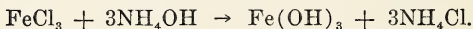
If it dissolves, *copper* is present. The solution will be blue, owing to the presence of copper nitrate,—



To confirm, add a little ammonium hydroxide to the original solution. A light blue precipitate forms. Add an excess of ammonium hydroxide. The precipitate dissolves to form a dark blue solution.

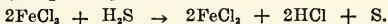
(ii) If the precipitate is orange, dissolve it by heating with hydrochloric acid. In the solution place a little pure zinc in contact with a piece of platinum foil. A black stain on the platinum is *antimony*.

4. If no precipitate has so far been obtained, boil the solution to expel hydrogen sulphide, and then boil again with a few drops of nitric acid. Add ammonium chloride, and then ammonium hydroxide in excess. A brown precipitate indicates *iron*, a white gelatinous one *aluminium*.
E.g.



(a) If hydrogen sulphide were not expelled, zinc (if present) would be precipitated as sulphide as soon as ammonia was added, because zinc sulphide is insoluble in an alkaline solution.

- (b) Nitric acid is added to oxidize any ferrous salt that may be present to ferric, because though a ferrous salt is precipitated by ammonia, the precipitation is not very complete. Even if the original salt were ferric, it will have been reduced to ferrous by the hydrogen sulphide. *E.g.*



- (c) Ammonium chloride is added to prevent the precipitation of magnesium at this stage.
- (d) It is easy to tell when ammonia is in excess, because after shaking, the solution will smell of the gas. When shaking, turn the test-tube away from yourself and other people, for the liquid often spurts out.

(iii) To find whether the original salt was ferrous or ferric, notice the colour of the original solution. A ferrous salt gives a water-green solution, a ferric salt a yellow or brown one.

The two following tests should also be tried.

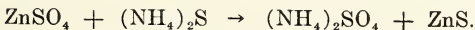
1st. To the original solution add freshly-prepared potassium ferricyanide¹. A deep blue precipitate of ferrous ferricyanide ("Turnbull's blue") means that *ferrous* ion is present.

2nd. To the original solution add ammonium thiocyanate. A dark red solution shows the presence of *ferric* ion.

With a ferrous salt there is no colour change. A *faint* pink may be disregarded. It means that a trace of ferric salt has been produced by atmospheric oxidation.

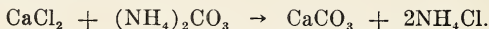
To confirm aluminium, filter, and wash the gelatinous precipitate with boiling water. Put a little on a charcoal block and heat with a blowpipe. Then moisten with a few drops of cobalt nitrate solution and heat again. A blue mass indicates *aluminium*.

5. To a portion of the alkaline solution obtained in Expt. 4 add ammonium sulphide. A white precipitate (coloured yellow with the ammonium sulphide) indicates *zinc*. *E.g.*



¹ *I.e., a solution* of potassium ferricyanide and so throughout this Exercise (and the next). If solid is intended, it will be definitely stated except where there could be no ambiguity (*e.g.* copper).

6. To another portion of the alkaline solution obtained in Expt. 4 add ammonium carbonate. A white precipitate indicates *calcium*. *E.g.*

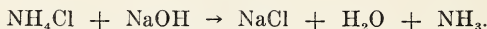


To confirm, apply the flame test, (Ex. 50), either to the precipitate (after filtering) or to the original substance. A brick-red coloration indicates *calcium*.

7. To the substance obtained in Expt. 6 add disodium phosphate. A white precipitate indicates *magnesium*.

The precipitate is magnesium ammonium phosphate, $\text{Mg}(\text{NH}_4)\text{PO}_4$.

8. Heat the original solution with sodium hydroxide. If ammonia is evolved (characteristic smell, and turns damp red litmus paper blue) the substance was an ammonium salt. *E.g.*



9. If the original substance was supplied in solution boil a little of the latter to dryness. Moisten the solid with a drop or two of dilute hydrochloric acid, and then apply the flame test. A *strong* golden-yellow colour indicates *sodium*.

A lilac colour (reddish violet when examined through blue glass) indicates *potassium*.

The part played by the blue glass is explained in Ex. 50.

Do not conclude that sodium is present unless (i) the yellow colour is really well-marked, and (ii) you are sure that no other base is present.

EXERCISE 52¹

QUALITATIVE ANALYSIS — ANIONS

Object.—To learn how to identify the acid radical or anion present in a given soluble salt.

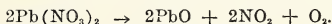
It is understood that the anion is one of the following:— OH^- (hydroxide), Cl^- (chloride), S^{--} (sulphide), PO_4^{---} (phosphate), SO_3^{--} (sulphite), SO_4^{--} (sulphate), CO_3^{--} (carbonate), NO_3^- (nitrate).

¹ See footnote p. 88.

Apparatus and Materials.—(a) *Bunsen; tripod stand; wire gauze; evaporating dish; rack of test-tubes.* (b) *Litmus paper; usual reagents for qualitative analysis; chlorides, phosphates, etc., for identification (see list of anions mentioned above).*

Directions, etc.—1. If the salt is supplied in solution, boil about half of the latter down to dryness, as the solid is needed for some of the tests.

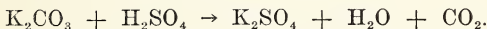
Take care not to heat the dry residue strongly, because various chemical changes may thereby take place. *E.g.* a nitrate would be decomposed,—



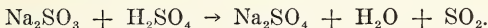
2. Test the solution with litmus paper. If the latter turns blue, hydroxide ion, OH^- , is present.

We must not conclude, however, that the substance in solution is necessarily a hydroxide. It may be a salt in which a strong base is combined with a weak acid (*e.g.* sodium carbonate).

3. Add dilute sulphuric acid, and afterwards warm. Violent effervescence in the cold (carbon dioxide evolved) indicates a *carbonate*. *E.g.*



Little action in the cold but effervescence on warming, with evolution of sulphur dioxide, indicates a *sulphite*. *E.g.*



(We recognize sulphur dioxide by its characteristic choking smell. Further, if a piece of filter paper is dipped in potassium dichromate and held in the mouth of the test-tube, it is turned green.)

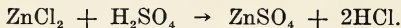
Sometimes in the cold, sometimes on warming, hydrogen sulphide is evolved (smells of bad eggs, and turns a filter paper dipped in lead acetate solution to a glossy black). If this happens a *sulphide* is present.

4. Add pure sulphuric acid and afterwards warm, but do not boil.

Boiling causes the production of thick, acrid fumes, and this would make it impossible to recognize any gases produced by the action of the acid on the salt. Pure acid is used because the “commercial” product often smells of sulphur dioxide.

Carbonate, sulphite and sulphide will give reactions very similar to those just described.

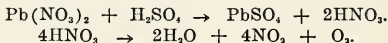
Effervescence with production of hydrogen chloride indicates a *chloride*. *E.g.*



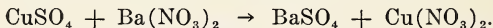
Hydrogen chloride is recognized by its characteristic pungent smell, and especially by a steamy appearance outside the test-tube, much increased by breathing across the mouth of the tube.

Faint brown fumes on heating (much more perceptible on looking down into the test-tube, but hold the latter at arm's length) indicate a *nitrate*.

Nitric acid is produced in the first instance, and a little of it is decomposed by the heat. *E.g.*



5. Add barium nitrate. A white precipitate insoluble in dil. hydrochloric acid indicates a *sulphate*. *E.g.*



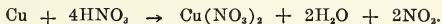
- (i) Barium chloride is often used instead of the nitrate, but a white precipitate in this case might be due to the presence of a silver salt or lead salt, giving insoluble silver chloride or lead chloride.
- (ii) It is important to make sure that the precipitate is insoluble in hydrochloric acid. Carbonates, sulphites, and phosphates will all give precipitates, but these are soluble.

6. Taking *one or two drops only* of the solution, add a solution of ammonium molybdate in nitric acid, afterwards warming gently. A fine yellow precipitate indicates a *phosphate*.

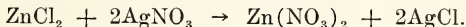
7. If no definite result has been obtained so far, it is most likely that either a nitrate, a sulphite or a chloride has been overlooked. Try additional tests for these as described in (8), (9), and (10).

8. Mix a little of the dry salt with copper filings, add a few drops of sulphuric acid and warm. Reddish brown fumes indicate a *nitrate*.

Thus with lead nitrate, nitric acid is first produced as already described in (3), and this is then reduced by the copper.

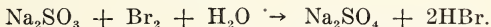


9. Dissolve a little of the salt in dilute nitric acid (or add this acid, if the salt is already in solution), and then add silver nitrate. A white precipitate, insoluble in ammonia, indicates a *chloride*. *E.g.*



10. To test for a sulphite, dissolve in water and then heat with a little bromine water. This will oxidize any sulphite present to a sulphate, which will now give the reaction described in (4).

E.g. the oxidation of sodium sulphite would be represented by the equation



Tabular Scheme.—In the foregoing account it has been necessary to introduce a number of explanations, equations, etc. in order that the student may be able to acquire an intelligent knowledge of what he is doing. There is some danger, however, that the introduction of such material may tend to obscure the general outline, and to meet this difficulty a tabular scheme will now be given for the identification of the “metal”, and another for the “acid”. Here there will be, purposely, only a bare minimum of information, but the student who wishes to make good progress in chemistry will find time to study the fuller explanations given in the preceding pages.

Tabular Scheme for examination of a Solution containing ONE of the ions

Ag^+ , Pb^{++} , Hg^+ , Hg^{++} , Cu^{++} , Sb^{++++} , Fe^{++} , Fe^{+++} , Al^{+++} , Zn^{++} , Ca^{++} , Mg^{++} , Na^+ , K^+ , NH_4^+ .

A. To the solution add a few drops of dil. HCl. If this gives a pp., add more acid until precipitation is complete. Examine according to I.

I. (Salts of Ag, Pb, Hg+.)	B. If no pp. obtained with HCl, pass H_2S through the acidified solution. If a pp. is obtained, examine according to II.	
Divide pp. just obtained into two parts. Add NH_4OH to one part and dil. HNO_3 to the other. Shake.	II. (Salts of Hg^{++} , Cu, Sb.)	C. If no pp. obtained so far, boil to expel H_2S , boil again with a few drops HNO_3 , add NH_4Cl and then excess of NH_4OH . If a pp. is obtained examine according to III.
(a) Pp. dissolves in NH_4OH but not in HNO_3 .— <i>Silver</i> .	Filter, wash with boiling water, and transfer a little of pp. to test-tube.	D. If no pp. obtained so far, add ammon. sulphide to portion of alkaline solution obtained in C. White pp. (coloured yellow with ammon. sulphide).— <i>Zinc</i> .
(b) Pp. goes grey-black on adding ammonia, but does not dissolve.— <i>Mercury (-ous)</i> .	(a) If pp. is black it is CuS or HgS . Boil with dilute nitric acid. Pp. insol.— <i>Mercury (-ic)</i> . Pp. sol.— <i>Copper</i> . Confirm as on p. 90.	E. If no pp. obtained so far, add ammonium carbonate to another portion of alkaline solution obtained in C. With pp.— <i>Calcium</i> . Confirm as on p. 92.
(c) To original solution add K_2CrO_4 . Yellow pp.— <i>Lead</i> .	(b) If pp. is orange-coloured — <i>Antimony</i> . Confirm as on p. 90.	F. If no pp. obtained so far, add disodium phosphate to solution from E. (a) White pp.— <i>Magnesium</i> . (b) If no pp. obtained so far, (i) heat original substance, or solution, with NaOH. Ammonia evolved.— <i>Ammonium</i> . (ii) Apply flame test to original substance. <i>Strong</i> golden yellow colour.— <i>Sodium</i> . Lilac colour (crimson when seen through blue glass).— <i>Potassium</i> .

Tabular Scheme for examination of a solution containing Carbonate, Sulphite, Sulphide, Chloride, Nitrate, Sulphate, or Phosphate.

Preliminary Operation.—Boil about half of the solution down to dryness, as the solid is needed for some of the tests.

Identification of	Method
Carbonate	To a little of the solid, add dilute sulphuric acid, and afterwards warm.
Sulphite	(a) Violent effervescence in the cold (CO_2 evolved)— <i>Carbonate</i> .
Sulphide	(b) Little action in the cold, SO_2 evolved on warming (smell, and action on potassium dichromate)— <i>Sulphite</i> . (c) H_2S evolved (smell, and action on lead acetate paper)— <i>Sulphide</i> .
Chloride	To a little of the solid, add sulphuric acid and afterwards warm, but do not boil.
Nitrate	(a) HCl evolved (characteristic pungent smell, steamy fumes)— <i>Chloride</i> . (b) Faint brown fumes on heating— <i>Nitrate</i> . [(c) Carbonates, sulphites and sulphides give reactions very similar to those obtained with dil. acid.]
Sulphate	Add barium nitrate. (a) White pp., insol. in dil. HCl — <i>Sulphate</i> . [(b) White pp. sol. in dil. HCl would be carbonate, sulphite or phosphate.]
Phosphate	Taking <i>one or two drops only</i> of the solution, add ammonium molybdate with nitric acid, afterwards warming gently. Fine yellow pp.— <i>Phosphate</i> .
Sulphite	(a) Heat with a little bromine water, then add barium nitrate. White pp. insol. in dil. HCl indicates that a <i>sulphite</i> was originally present (oxidized by the bromine water to a sulphate).
Chloride	(b) Add dil. HNO_3 , then AgNO_3 . White pp. sol. in ammonia— <i>Chloride</i> .
Nitrate	(c) Mix a little of the dry salt with copper filings, add a few drops of sulphuric acid, and warm. Reddish brown fumes— <i>Nitrate</i> .
(Additional Tests)	

APPENDIX I

FIRST AID TREATMENT

N.B.—Many of the accidents which may happen in the chemical laboratory are such as often happen elsewhere, *e.g.* burns, scalds, and cuts. The few suggestions which follow deal, however, with accidents which seldom happen outside a laboratory, and the first aid treatment of which is less likely to be familiar.

1. *Acid on hands.* See no. 4 of "Safety First" rules.

2. *Acid splashes on face.* A 3% solution of sodium bicarbonate should be squirted on from a wash-bottle.

If any acid has entered the eye the treatment must be very prompt (the solution just mentioned can be squirted into the eye without injury).

Such a wash bottle should *always* be kept in a very accessible place, and clearly labelled "*Sodium bicarbonate solution. To be squirted into the eye at once if any acid has entered. On no account to be tampered with or removed.*"

Boys are very apt to fool about with a wash-bottle, but it is the writer's experience that they always respect this particular one if its purpose is explained to them.

3. *Alkali splashes on face (e.g. caustic soda).* A wash-bottle containing a 1% solution of acetic acid would be the ideal resource, but in practice the bicarbonate solution serves very well.

4. *Accidents with large quantities of acid (e.g. the result of breaking a bottle of acid).* Water should be used in large quantities. The usual "fire bucket" is excellent in most cases, but it should be somebody's regular job to keep it filled. After the bulk of the acid has been washed away, sodium bicarbonate may be dusted over the injured surface, to neutralize any acid that still remains. The wound may then be treated as an ordinary burn.

5. *Phosphorus burns.* Wash well with water so that no phosphorus is left in the wound, and then sponge with a piece of lint soaked in dilute silver nitrate solution. (Use solution from reagent bottle diluted with three times its bulk of water). Then put a tight bandage over, to exclude all air.

Questions

What would you do if —

1. Your neighbour were mixing two or three chemicals together “just to see what would happen”?
2. You wished to use a solution of caustic soda, but found that the glass stopper was tightly stuck?
3. At the close of the lesson it was necessary to wash out a flask which had contained hot sulphuric acid?
4. When preparing hydrogen chloride, you saw sulphuric acid rising rapidly in the thistle funnel of the generating flask?
5. You noticed that another student when heating a test-tube had a persistent habit of pointing it in your direction?
6. You wished to push a thistle funnel through a rubber stopper, but found it was a very tight fit?
7. You had spilt some nitric acid on your hand?
8. A bottle of sulphuric acid had slipped out of your hand, and a quantity of it had spilt over your foot?
9. You saw that a neighbour had incautiously sniffed at a jar of chlorine, and appeared to be in great pain?

APPENDIX II

“Apparatus and Materials.—(a)”

(i) Apparatus

3 beakers (about 250 c.c.)	Enamel dish ¹
2 evaporating dishes (85 c.c. and 225 c.c.)	Clamp and boss (“collar”)
	Crucible tongs (6”)
Rack with 11 test-tubes (6” × 5/8”) and 1 boiling tube (6” × 1”)	Pipeclay triangle (2” side)
	Glass stirring rod
	Test-tube brush
Retort stand 18” high, with 2 rings (3” and 2” internal diam.)	Tin tray (sand-bath), 6” diam.
	Wire gauze 4” square
Tripod stand	Taper
Beehive shelf	Bunsen, with 2 feet of rubber tubing (on bench)
Funnel (3” diam.)	
Deflagrating spoon	

(ii) Materials (Reagents)

Sulphuric acid ²	Sulphuric acid dilute ³
Hydrochloric acid ²	Hydrochloric acid dilute ³
Nitric acid ²	Nitric acid dilute ³
Calcium hydroxide	Ammonium hydroxide ³

N.B.—When in the various exercises “sulphuric acid” is mentioned, the *concentrated* acid is to be understood. If dilute acid is intended, the word “dilute” will be used; and similarly for the other acids. “Ammonium hydroxide” will indicate the diluted solution mentioned above.

¹ A cheap dish which is put on the bench at the beginning of each lesson to receive used matches, used filter papers, etc. It is emptied into the waste-box at the end. It greatly reduces the tendency to throw paper, etc., into the sink.

² These are “concentrated”, i.e. sulphuric acid sp.gr. 1.84, hydrochloric acid sp.gr. 1.16, and nitric acid sp.gr. 1.42.

³ These are of strength about 5*N*. A sufficiently close approximation for general purposes is obtained as follows:—

Sulphuric acid. Water 6 volumes, acid (sp.gr. 1.84) 1 vol.

Hydrochloric acid. Water 1 volume, acid (sp.gr. 1.16) 1 vol.

Nitric acid. Water 11 volumes, acid (sp.gr. 1.42) 5 vols.

Ammonium hydroxide. Water 3 volumes, ammonia solution (sp.gr. 0.88) 1 vol.

APPENDIX III

REAGENTS MOST COMMONLY USED IN ANALYSIS

When doing qualitative analysis, all pupils will require the following reagents besides those mentioned in Appendix II:—

Ammonium carbonate. Dissolve 200 gm. of the commercial "carbonate" (sal volatile) in 350 c.c. of 5*N* ammonia and make up to 1 litre.

Ammonium chloride. 267 gm. per litre (5*N*).

Ammonium sulphide. Take 600 c.c. of 5*N* ammonia and saturate this with hydrogen sulphide. Then make up to 1 litre; or the solution as usually bought may be diluted with an equal volume of water.

Barium chloride. 122 gm. per litre (*N*).

Barium nitrate. 65 gm. per litre (*N*/2).

Disodium phosphate. 119 gm. per litre (*N*).

Hydrogen sulphide solution. (If Kipp's apparatus is not available),—a saturated solution.

Silver nitrate. 34 gm. per litre (*N*/5).

Sodium hydroxide. 200 gm. per litre (5*N*).

APPENDIX IV

SPECIAL REAGENTS

N.B.—Unless something is said to the contrary, the solutions mentioned under “Apparatus and Materials.—(b)” are of the strength indicated below, and so may be taken from this “Special Reagent” shelf.

Acetic acid. To 1 vol. of the glacial acid add $2\frac{1}{2}$ vols. of water (5N).

Ammonium molybdate. (i) Dilute 50 c.c. of “0.88” ammonia with an equal volume of water, and dissolve 35 gm. of crystallized ammonium molybdate in the liquid.

(ii) Add 180 c.c. of nitric acid to an equal volume of water.

(iii) Slowly add the solution obtained in (i) to that obtained in (ii), keeping the liquid cool while mixing. Let the mixture stand for 24 hours.

(iv) Decant the liquid from any molybdic acid which may have separated, and dilute it with an equal volume of water.

Ammonium oxalate. 40 gm. per litre (N/2).

Ammonium thiocyanate. 15 gm. per litre (N/5).

Calcium sulphate. A saturated solution (N/30).

Chlorine water. Pass chlorine through water till saturated (N/5). Fresh solution has to be prepared rather frequently.

Cobalt nitrate. 73 gm. of the crystallized salt per litre (N/2).

Ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. 90 gm. per litre (N).

Lead acetate. 190 gm. per litre (N). To obtain a clear solution it is necessary to add a little acetic acid, which prevents hydrolysis.

Litmus. Add hot water to about 10 gm. of solid litmus (powdered). Stir, allow to settle, and pour off the coloured liquid. Add more hot water to the residue, repeating the whole process two or three times. Now leave the coloured liquid for a few hours to secure complete settling, decant into a bottle and make up to about 200 c.c. Add a little very dilute nitric acid until the colour is just purple.

The stopper for a litmus bottle should fit only loosely,—just enough to keep out the dust. Litmus soon loses its colour if air is completely excluded.

Mercuric chloride. 27 gm. per litre (N/5).

Potassium chromate. 97 gm. per litre (*N*).

Potassium ferricyanide. 110 gm. per litre (*N*).

This solution soon undergoes change, and it is better to make up only a small quantity just before the lesson.

Potassium ferrocyanide. 105 gm. per litre (*N*).

Potassium iodide. 33 gm. per litre (*N/5*).

Stannous chloride, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$. Heat 112 gm. of the salt with 200 c.c. of dilute (*i.e.* *5N*) hydrochloric acid until a clear solution is obtained, and then make up to 1 litre with water (*N*). Put some fragments of granulated tin in the reagent bottle to prevent oxidation.

Starch. Mix about 1 gm. of starch into a paste with cold water, and pour the paste slowly into 200 c.c. of boiling water. To the latter add either 0.01 gm. of mercuric iodide or 1 gm. of salicylic acid. Allow to cool and settle, and decant the clear liquid into a bottle. If the mercuric iodide, etc., has been added the solution will keep for months.

The Reagent shelf should also contain the following solids:—

Manganese dioxide, borax, copper (powder or filings) and *ferrous sulphate* (fine crystals).

APPENDIX V

SUPPLIES FOR 10 STUDENTS

The following is a list of the supplies required by 10 students working singly, or 20 working in pairs, for the exercises in this Manual. A reasonable margin has been allowed for breakages, etc., and the fact has been kept in mind that it is usually cheaper to buy articles by the dozen, or (in the case of chemicals) by the pound or half-pound. The list is sub-divided into—

- A. Apparatus required by all.
- B. Apparatus for general use (*e.g.* barometer).
- C. Apparatus desirable, though not absolutely essential.
- D. Chemicals.
- E. Materials best obtained locally.

A. Apparatus required by all

Beakers ¹ , 250 c.c.	4 doz.	Glass tubing, ext. diam.	
Beehive shelves ² , 3"×1½"	1 doz.	6 mm.	4 lb.
Blowpipes	1 doz.	Glass tubing, ext. diam.	
Blue glasses, 2"×1"	1 doz.	5-5½ mm.	1 lb.
Bunsens or alcohol lamps	1 doz.	Glass tubing, ext. diam.	
Burettes, 50 c.c., to show 0.1 c.c.	1 doz.	6½-7 mm.	1 lb.
Burette stands ³	10	Graduates, 100 c.c.	1 doz.
Charcoal blocks (com- pressed), 6"×1"×1"	1 doz.	Labels, blank gummed	1 packet (100)
Clamp (with boss-heads)	10	Measuring flasks, 250 c.c.	1 doz.
Combustion tubes, 12"× ⅝" int.	1½ doz.	Non-combustible rods for flame tests	1 box (100)
Cover slips, 3" diam.	5 doz.	Pipeclay triangles, 2" side	1 doz.
*Crucibles, nickel, 3 cm. diam.	1 doz.	Pipettes, 25 c.c.	1 doz.
Crucible tongs, 6"	1 doz.	Platinum wire for flame tests, or non-combustible rods (<i>q.v.</i>) as substitute	2 ft.
Deflagrating spoons	1 doz.	Pneumatic troughs, earthen- ware, 12" diam.	1 doz.
Dishes, cheap enamel, about 7½"×2"	1 doz.	Retorts, 250 c.c.	1 doz.
Evaporating dishes, 85 c.c.	1 doz.	Retort stands, 18" high, with 2 rings of 3" and 2" int. diam.	1 doz.
do. 225 c.c.	1 doz.	Rubber stoppers, diam. narrow end 9/16ths,	
Files, triangular, 5"	10	1 - hole	2 doz. ⁴
Filter papers, 5" diam.	1000	Rubber stoppers, diam. narrow end 15/16ths,	
Flame spreaders	10	2 - hole	2 doz. ⁵
Flasks, 500 c.c., flat bot- tom, int. diam. of neck 1"	1½ doz.	Rubber tubing, for bunsens	24 ft.
Flasks, 500 c.c., conical, neck as above	1 doz.	Test-tubes, 6"×⅝ths	1½ gross
Funnels, 3" diam.	1 doz.	do. 6"×1"	3 doz.
*Funnels, separating: 150 c.c.	1 doz.	do. 6"×⅝ths, hard glass	3 doz.
Gas jars, 8"×2" (int.)	4 doz.	Test tube brushes, bristle end	1 doz.
Glass rod, 6mm. diam.	2 lb.		

¹ Beakers, flasks and test-tubes made of a "resistance glass" such as Monax, Pyrex, etc. cost more, but are cheaper in the long run.

² Unless the shallower type is asked for, they are often supplied too deep.

³ Not quite essential; a retort stand, with collar and clamp, serves fairly well.

⁴ To fit test tubes and combustion tubes.

⁵ To fit flasks and large size test-tubes.

Test-tube racks, 12 holes and pegs	10	Thistle funnels, heads only ¹	1 doz.
*Thermometers, —10°C. to 110°C., narrow stem for passing through corks	1 doz.	Tin trays (sand baths) 6" diam.	1 doz.
*Thermometers, —10°C. to 50°C., by 0.2°	1 doz.	Tripod stands	10
Thistle funnels, 12", ext. diam. of stem 6 mm.	1½ doz.	Watch glasses, 5 cm.	1 doz.
		Wire gauze squares, iron, 4" side	2 doz.
		Wooden splints	2 bundles

B. Apparatus for General Use

Balances, with weights	2	Mortars and pestles, 5¼" ext. diam.	3
Barometer	1	Mortars and pestles, 10" ext. diam.	1
Cork-borers	1 set	Mortars and pestles, 6" ext. diam., iron	1
Desiccators, 8"	2		
Kipp's apparatus for H ₂ S	2		

C. Apparatus desirable, though not absolutely essential

Drying oven (air), 9"×9"×9"	Measuring flasks, 100 c.c., 500 c.c. and 1 litre
Foot blow-pipe	Muffle furnace, to take a muffle 8"×4¼"×3¼"
Liebig's Condenser, 28" over all	

D. Chemicals

*Acetone	½ lb.	Ammonium sulphide solution	2 W. qts.
Alcohol, absolute	4 oz.	Ammonium thiocyanate pure	2 oz.
Alcohol, denatured	2 lb.	Antimony metal, regulus	1 lb.
*Alcohol, methyl (wood spirit)	½ lb.	Antimony trichloride cryst.	¼ lb.
Alum, pure powder	1 lb.	Arsenious oxide	¼ lb.
Aluminium foil	2 oz.	Barium chloride pure	1 lb.
Aluminium sulphate, pure cryst.	¼ lb.	Barium nitrate pure	1 lb.
Ammonia, 0.88	3 Winchester quarts (about 13½ lb.)	Bismuth trichloride	2 oz.
Ammonium carbonate powder, resub.	1 lb.	Bleaching powder	1 lb.
Ammonium chloride cryst.	2 lb.	Borax cryst.	½ lb.
do. purest	1 lb.	Bromine	½ lb.
		*Calcium carbide	1 lb.

* Required for exercises marked with an asterisk.

¹ Gradually accumulated; or a dealer will often supply them very cheaply from his own damaged stock.

Calcium chloride, pure cryst.	1/2 lb.	Methyl orange	1/2 oz.
*Calcium fluoride (fluor- spar powder)	1/2 lb.	Nitric acid coml.	2 W. qts
*Calcium sulphate, pure precip.	1/4 lb.	do. pure	1 W. qt.
*Camphor	1/2 oz.	*Paraffin wax, m.p. 54°C.	1/4 lb.
Charcoal, animal	1/2 lb.	Phenolphthalein	1/2 oz.
Chloroform (not purest)	1/2 lb.	Potassium bromide pure	1 1/2 lbs.
Copper filings	1 lb.	*Potassium chlorate, pure cryst.	1/2 lb.
Copper foil	1/2 lb.	Potassium chloride pure.	1 lb.
Copper sulphate, coml. cryst.	3 lbs.	Potassium dichromate pure pure cryst.	1/2 lb.
Copper sulphate, pure cryst.	1 lb.	Potassium ferricyanide pure cryst.	1/2 lb.
Cupric bromide	2 oz.	Potassium ferrocyanide pure cryst.	1/2 lb.
Cupric chloride, pure cryst.	1 lb.	*Potassium hydroxide (pellets)	1 lb.
Ferric chloride pure	1 lb.	Potassium iodide	1/2 lb.
Ferrous sulphate pure	1 lb.	Potassium nitrate pure cryst.	1 lb.
Ferrous sulphide, plates	5 lb.	Potassium permanganate cryst.	1 1/2 lb.
Glucose, coml.	1 lb.	Silver nitrate, pure recryst.	4 oz.
do. pure anhyd.	1/4 lb.	*Soap, Castile (powder)	1 oz.
Hydrochloric acid coml.	4 W. qts.	*Sodium acetate fused	1 lb.
do. pure	2 W. qts.	Sodium bicarbonate, pure powder	2 lb.
Iodine resub.	2 oz.	Sodium carbonate, pure cryst.	3 lb.
Iron filings	1 lb.	Sodium chloride pure	1 lb.
Lead foil	1 lb.	Sodium hydroxide, white sticks, ordinary	3 lb.
Lead nitrate, pure cryst.	1 lb.	Sodium nitrate coml.	1 lb.
Lead peroxide coml.	1/4 lb.	do. pure cryst.	1 lb.
Lead (red)	1 lb.	Sodium peroxide	2 oz.
Litharge, powder	1/4 lb.	Sodium (disodium) phos- phate, purest	1 lb.
Lithium chloride	1/2 oz.	Sodium sulphite cryst.	1 lb.
Litmus, best quality gran.	2 oz.	Sodium thiosulphate	1 lb.
Litmus books, neutral	2 doz.	Starch	1 lb.
Magnesium, ribbon	1 oz.	Strontium chloride, pure cryst.	1 oz.
Magnesium carbonate, heavy	1/2 lb.	Sulphuric acid coml.	4 W. qts.
Magnesium chloride, pure cryst.	1/2 lb.	do. pure	1 W. qt.
Magnesium sulphate, pure cryst.	1/2 lb.		
Manganese dioxide, coml. powder	2 lb.		
Mercury	1/2 lb.		
Mercuric chloride, gran.	1/4 lb.		
Mercurous nitrate	2 oz.		

*Tallow	2 oz.	Zinc foil	¼ lb.
*Urea	¼ lb.	Zinc sulphate, pure cryst.	1 lb.
Zinc (free from As)	1 lb.	Zinc sulphide compl.	½ lb.

E. Materials best obtained locally

*Bone	A few oz.	Marble chips	A few lb.
Clay	About 1 lb.	Medicine bottles (about 6 oz.), with corks	10
Clock-spring	About a yard in all	Molasses, black	½ lb.
Distilled water	1 gal.	Red calico	¼ yd.
Finger bandaging 2" wide	6 yds.	Salt, (coarse, for freezing mixtures)	5 lb.
Glass, sheets of, about 4"×4"	10	Sand	A few lb.
Honey	1 oz.	Soot	A few oz.
Ice, as required		Sugar	½ lb.
Iron nails, about 1", bright	10	Thread	1 reel
*Lard	2 oz.	*Wood ash	About 2 lb.
Lime (quicklime)	A few lb.	*Yeast	4 oz.
*Malt extract (<i>e.g.</i> Liebig's)	1 oz.		

APPENDIX VI

MAXIMUM PRESSURE OF AQUEOUS VAPOUR (IN MM. OF MERCURY)

°C	mm.	°C	mm.	°C	mm.	°C	mm.
5	6.5	11	9.8	17	14.4	23	20.9
6	7.0	12	10.4	18	15.3	24	22.2
7	7.5	13	11.1	19	16.3	25	23.5
8	8.0	14	11.9	20	17.4	26	25.0
9	8.5	15	12.7	21	18.5	27	26.5
10	9.1	16	13.5	22	19.6	28	28.1

APPENDIX VII

A SIMPLE RACK FOR GAS-GENERATING FLASKS

THE drawing will almost explain itself. The rack is made of wood $\frac{5}{8}$ " — $\frac{3}{4}$ " thick, and is secured to the wall with brackets. Between the lower shelf and the bench there is a clearance of $8\frac{1}{2}$ inches. The space that takes the neck of the flask can be closed by a simple hook which prevents the flask from falling out.

Each flask is connected with a double-bend delivery tube, the latter being just held at G by a simple wire grip. A right-angled delivery tube is often wanted, and this passes through the holes P, Q.

The rack can, of course, be of any length. The one used in the writer's laboratory takes 15 flasks.

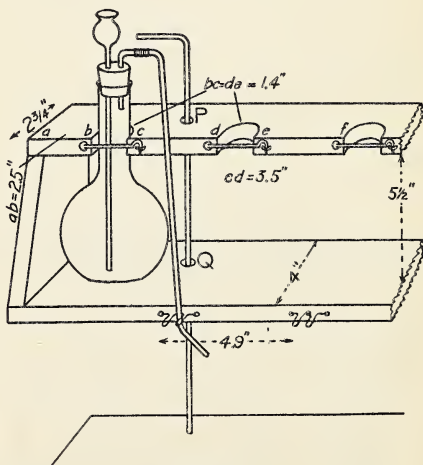


FIG. 25.

APPENDIX VIII

Table of Solubilities

	Bromide	Carbonate	Chlorate	Chloride	Hydroxide	Iodide	Nitrate	Phosphate	Silicate	Sulphate	Sulphide
Aluminium...	W	..	W	W	A	W	W	A	A	W	A
Ammonium...	W	W	W	W	W	W	W	W	..	W	W
Barium.....	W	A	W	W	W	W	W	A	A	Ins.	W
Calcium.....	W	A	W	W	W	W	W	A	A	W	W
Copper.....	W	A	W	W	A	W	W	A	A	W	A
Hydrogen....	W	W	W	W	W	W	W	W	W	W	W
Iron (ferrous).	W	A	W	W	A	W	W	A	A	W	A
Iron (ferric)...	W	..	W	W	A	W	W	A	A	W	A
Lead.....	A	A	W	A	A	W	W	A	A	A	A
Magnesium...	W	A	W	W	A	W	W	A	A	W	A
Nickel.....	W	A	W	W	A	W	W	A	A	W	A
Potassium....	W	W	W	W	W	W	W	W	W	W	W
Silver.....	Ins.	A	W	Ins.	..	Ins.	W	A	..	W	A
Sodium.....	W	W	W	W	W	W	W	W	W	W	W
Zinc.....	W	A	W	W	A	W	W	A	A	W	A

NOTES

- W** = Sol. in water; **W** = Sparingly sol. in water.
A = Sol. in one of the common acids (usually dil. HCl or HNO₃);
A = Sparingly sol. in acid.
Ins. = Insoluble in water and acid.
- It is worth while *remembering* that the following are soluble:—
 - All common compounds of sodium, potassium and ammonium.
 - All chlorides, except those of silver, lead and mercury (-ous).
 - All nitrates and chlorates.
 - All sulphates, except those of lead, barium, strontium and calcium (the last very slightly soluble).
- Carbonates and phosphates, except those of sodium, potassium and ammonium (*cf.* above) are *insoluble*.

Commoner Elements: their Atomic Weights and Valencies

Element	Symbol	Atomic Wt. (approx.)	Atomic Wt. (more exact)	Valence
Aluminum	Al	27	26.97	3
Antimony (stibium) .	Sb	122	121.77	3, 5
Argon	A	40	39.91	0
Arsenic	As	75	74.96	3, 5
Barium	Ba	137	137.37	2
Bismuth	Bi	209	209.00	3
Boron	B	11	10.82	3
Bromine	Br	80	79.92	1
Cadmium	Cd	112	112.41	2
Calcium	Ca	40	40.07	2
Carbon	C	12	12.00	4, 2
Chlorine	Cl	35.5	35.46	1, 5, 7,
Chromium	Cr	52	52.01	3, 6, 7,
Cobalt	Co	59	58.94	2, 3
Copper (cuprum) . .	Cu	64	63.57	2, 1
Fluorine	F	19	19.00	1
Gold (aurum) . . .	Au	197	197.2	1, 3
Helium	He	4	4.00	0
Hydrogen	H	1	1.008	1
Iodine	I	127	126.93	1
Iridium	Ir	193	193.1	3, 4, 2
Iron (ferrum) . . .	Fe	56	55.84	3, 2
Lead (plumbum) . .	Pb	207	207.2	2, 4
Lithium	Li	7	6.94	1
Magnesium	Mg	24	24.32	2
Manganese	Mn	55	54.93	2, 7, 4, 6
Mercury (hydrargyrum)	Hg	201	200.61	2, 1
Neon	Ne	20	20.2	0
Nickel	Ni	59	58.69	2, 3
Nitrogen	N	14	14.01	3, 5
Oxygen	O	16	16.00	2
Phosphorus	P	31	31.03	5, 3
Platinum	Pt	195	195.23	4, 2
Potassium (kalium) .	K	39	39.10	1
Radium	Ra	226	225.95	2
Selenium	Se	79	79.2	2, 6, 4
Silicon	Si	28	28.06	4
Silver (argentum) . .	Ag	108	107.88	1
Sodium (natrium) . .	Na	23	23.00	1
Strontium	Sr	88	87.63	2
Sulphur	S	32	32.06	2, 4, 6
Thorium	Th	232	232.15	4, 3
Tin (stannum) . . .	Sn	119	118.70	2, 4
Tungsten (wolframium)	W	184	184.0	6, 4, 2
Uranium	U	238	238.17	6, 4
Zinc	Zn	65	65.38	2

QD 33 L77 1934 LAB-MAN- C-2
LITTLER W
ELEMENTARY CHEMISTRY

39332528 CURR HIST



000005544945

CONTENTS

	Page		Page
7. Other Compounds of Iron	66	Appendix	
8. Copper and some of its Compounds	68	I. First Aid Treatment and Questions	98
9. Silver and some of its Compounds	70	II. " <i>Apparatus and Materials—(a).</i> " ..	100
10. Halogen Compounds of Silver	72	III. Reagents most commonly used in Analysis	101
11. Silver from a Coin	73	IV. Special Reagents	102
12. Oxides of Lead	74	V. Supplies for 10 Students	103
13. Other Compounds of Lead	76	VI. Maximum Pressure of Aqueous Vapour ..	107
* Acetylene	77	VII. A Simple Rack for Gas-generating Flasks ..	108
* Starch	79	VIII. Table of Solubilities	109
* Sugars	81	Commoner Elements: their Atomic Weights and Valencies	110
* Alcohol	82		
* Acetic Acid	84		
* Soap	85		
Flame Tests	87		
Qualitative Analysis-Cations	88		
Qualitative Analysis-Anions	92		

Cloth used in the binding of this book was produced especially for the publishers by Canadian Industries Limited. It is waterproof.

